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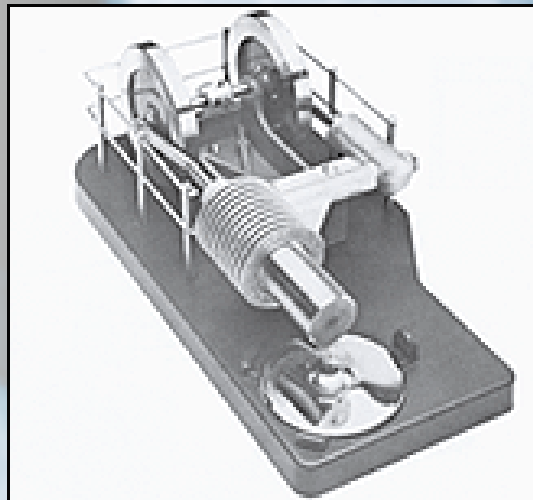


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MATERIALS

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Advanced Conductor Shape Technology

Оптимизиране формата на секция от правоъгълен меден проводник намираща приложение във статорите на хибридните двигатели

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Abstract: *The aim of the project is to analyze the possible section geometries of stators for starter generators and to select the most effective geometry for use in practice. The experimental results showed the benefits of the "S"-shape conductor compared with "P" or "U"-shape.*

Keywords: CONDUCTORS, HYBRID STATORS, BASKET OF STATOR.

Problem Relevance

The world's leading automotive manufacturers constantly thrive to improve fuel economy. This has taken automobile production in a new direction – the introduction of hybrid vehicle. It has been proven that a hybrid automobile can reduce fuel consumption by up to 50% when compared to conventional engine powered vehicle. The idea is not entirely new however its realizing has seen several major hurdles in the way: very high cost of production for the batteries and the requirement for highly effective (high capacity) starter-alternators. The rapid development of modern technologies managed largely to solve the problem with the batteries. Automotive manufacturers are currently working hard to develop highly efficient starter-generators. In order to increase the starter-generators efficiency, one must first optimize the stator design. This is where the possibilities of increasing efficiency are the greatest.

M3D, a machine manufacturing company, has vast experience in developing machines and technologies that have superior applications in the production of starters and generators. The company has produced machines which operate in some of the world's best automotive producers, such as Bosch and Valeo-Europe, Delco and Prestolite – USA, Denso and Mitsubishi – Japan. By combining the theoretical and experimental experience of the Institute of Metal Science, Equipment and Technologies with Hydro- and Aerodynamics Centre "Acad. A. Balevski" at the Bulgarian Academy of Sciences and with the technological experience of M3D, we have been able to create a setting for ultimate success with these developments.

The thesis of the following research is to create a conductor with new geometry which will improve the stator characteristics.

Problem analysis

The classic generator-stator with coils made of round conductors is technically obsolete, mainly due to the coefficient of filling the stator slot with a round conductor is smaller when compared to a conductor with a rectangular section. This is the reason why all hybrid stators nowadays use only rectangular conductors. Optimizing the conductor geometry of such conductors can indeed help increase the stator efficiency. The purpose of optimizing the geometry of the conductors is for them to form a basket (which is a collection of the conductor heads pointing in the opposite direction of the stator-side outlets/terminals) after being inserted in the stator core (body). The basket should have minimal dimensions, however, the hairpins shoulders should not come into contact with each other. The other condition that must be met is the distance $W1 \leq W3$ (fig. 1). With the upper limits in mind, it is necessary to optimize the shape of the conductors, so that the optimum density of the basket can be achieved, with maximum possible airflow between conductors. This requirement has been formed under one highly important condition – the need for achieving optimal cooling of the stator, for the purpose of reducing heat created in the stator, which can result in over 30% efficiency loss.

The current work process utilizes a 3-dimensional method of shaping the conductor head, i.e., realizing deformation along 3 axes – moving the pin, which allows bending along the X-axis, twisting along the plane of X and Y, as well as controlled positioning of the movable conductor shoulder in the direction of the Z-axis (fig 2.1). Applying deformation in the Z-axis is realized for the first time, but as the following results will show, it is of paramount importance. This deformation helps to control

the shoulders slope in the crown, and thus helps to achieve maximum airflow between the crowns of conductors, in other words the best cooling in the coil.

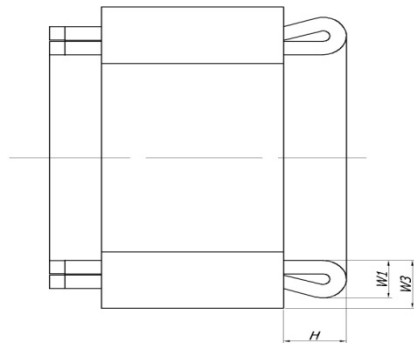


Fig.1

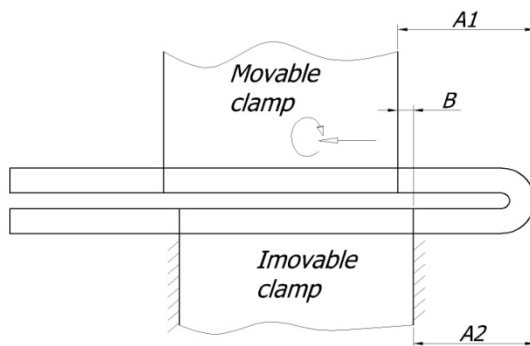


Fig.2.1

Theoretical and experimental methods of solving the problem

A combined approach was used in the experiment optimization – experiments paired with mathematical and computer methods for simulation. The entire forming process can be simulated with the help of specialized software. After which, full analysis of the advantages and disadvantages of each conductor shape can be made. For instance, for “U”-shape of semi-complete conductors, it can be visualized how the final desired form is achieved (fig. 2.2 and fig. 2.3). These results are obtained by experimentation as the mechanical characteristics for copper conductors with precise dimensions were entered in the software. The software also helped in achieving deformations, as well as the spread stress amount in every part of the conductor during the forming process. The

software further permits yet another step, which is inserting the deformed conductor in the stator body and to visualize the basket. This approach enables us to seeing the placement of the conductors in the stator slots, and to also check how they are arranged in the stator basket. This allows us to deduct whether the angle of twist in the last technological step needs to be adjusted and by how much. Following this sequence, the desired geometric parameters of the section are achieved after several iterations.

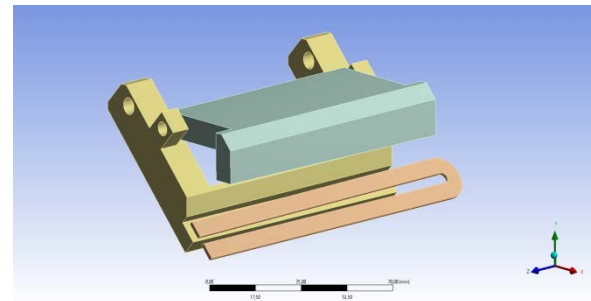


Fig.2.2

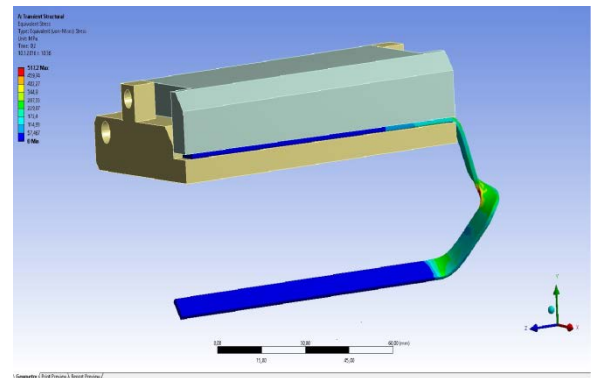


Fig.2.3

Experimental results

Practice shows that three important parameters related to the conductor geometry and forming the stator basket must be taken into consideration during the forming of the conductors.

The distance between two adjacent shoulders of conductors forming the basket. Figures 3 and 4 show the shoulder shape in the basket. In this case “P”-shape and “S”-shape can be seen. In the case of “P”-shape, the shoulders are part of the horde, whereas with “S”-shape, the shoulders are rainbow shaped. If the angle of twist of the “P”-shape is large enough, then there is a high possibility that the outside shoulder will touch the inside shoulder. This is the main reason for the high risk of shorting in this area. To avoid this problem, especially with bare conductors,

manufacturers that use “P”-shape sections are usually forced to place an insulating tape between the inside and outside conductor shoulders. This is a highly labour-intensive method and because of that has limited application.



“S”- form

Fig.3

Another way to avoid this problem is by increasing the pin diameter, where a process known as U-bending is applied. This, however, leads to a different problem – increasing the height of the crown ($H_1 > H$) (fig. 5). As of this reason, the idea has limited application, due to the likelihood of insufficient space. As Figure 5 shows, using an “S”-shape conductor is the solution. Several problems that require a solution occur in shaping the basket:

-Contact between two adjacent shoulders in the basket. This is a fairly common problem for conductors with small crown height. After twisting is applied, the

space between two neighbouring shoulders is usually larger in the outside row than on the inside row (fig. 6).



“P”- form

Fig.4

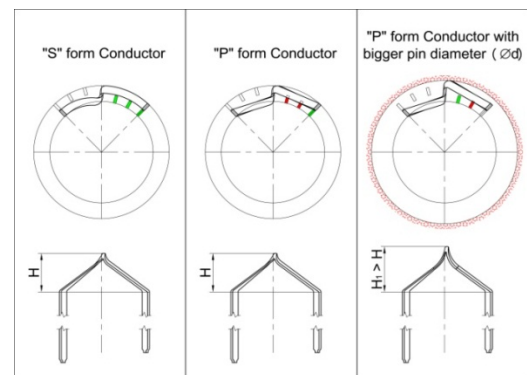


Fig.5

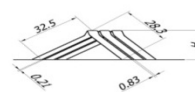


Fig.6

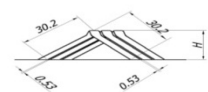


Fig.7

In order to achieve even distances between the adjacent inside and outside shoulders, it is necessary to alter the length of the shoulders in the crown by means of moving in the direction of Z-axis (fig. 2.1). These distances can be made equal by varying the magnitude of movement “B”, if all other parameters remain unchanged. It should be noted that this can be achieved without having to change the height of the crown “H”.

-Controlling the interior and exterior diameter of the stator basket. The possible amount of control is limited for “P” and “U”-shape conductors. It is possible with “S”-shape sections by shifting the section axis of symmetry in the direction of the X-axis (fig. 8).

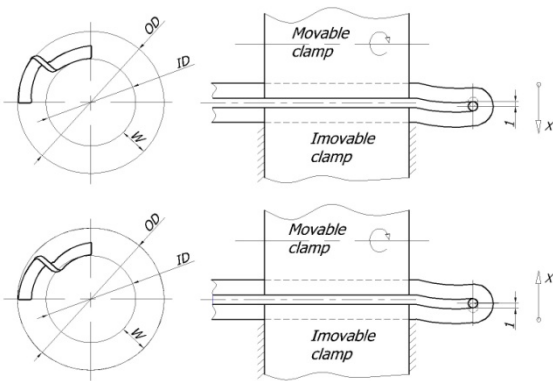


Fig.8

As the figure shows, if during the process of forming the pin around which the twisting occurs, it is positioned in the direction outside the axis of symmetry of the semi-complete conductor (for example, by 1mm as shown in the figure), the interior and exterior diameter of the basket will increase. If the pin is moved in the opposite direction, i.e. towards the axis of symmetry, then the interior and exterior diameter of the basket will decrease. Fine adjustments to the height of the basket can be made using that method. This cannot be applied in the case of “P”-shape, because the axis of symmetry and axis of rotation must coincide.

The production technology of “S”-shape (fig. 9) looks similar to that of “P”-shape, but only seemingly, which is apparent in the big difference in the final shape of the conductor crown. By changing the radii “R” and “r” in step 2, the desired arcs can be set. The above described steps can be visualized with the help of the software. This software can simulate the entire technological process, and thus can analyze the effect of every separate parameter involved in the forming of the conductor. As can be seen in the case of “P”-shape, the distance between the inside shoulders of two adjacent conductors is very small (fig. 10). This is the reason for probably shorting with “P”-shape. As the figure depicts, this effect is increased when the angle of twist grows. If the angle of twist is 20 degrees, the distance between the inside shoulders is sufficient, whereas if the

angle is increased to 45 degrees, the risk of shorting is much higher. The experimental results show that the problem is completely eliminated by utilizing “S”-shape conductors (fig. 11).

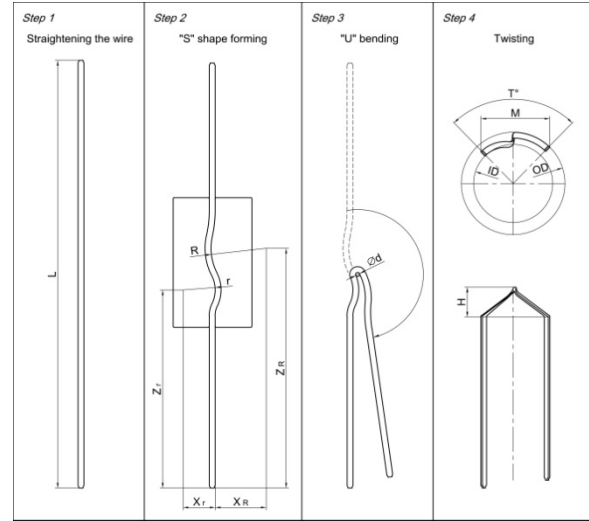


Fig.9

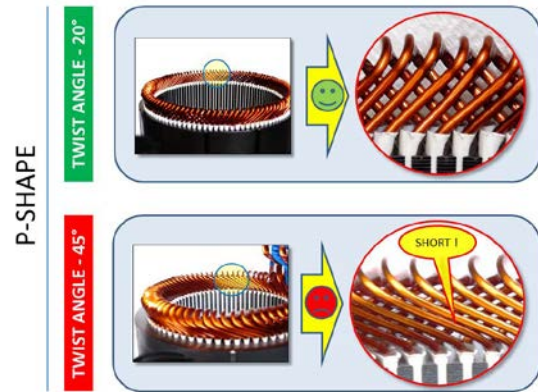


Fig.10

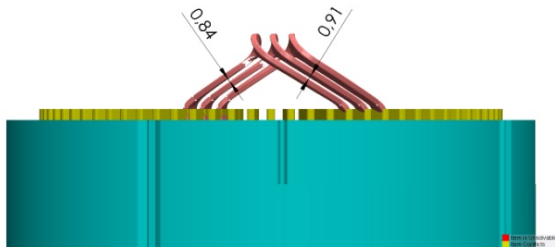
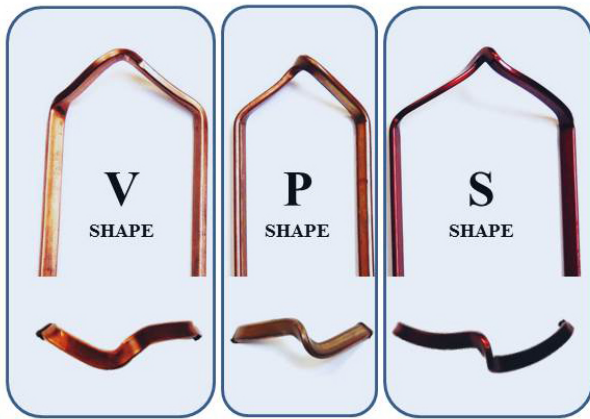


Fig.11



Analysis of the results

We will conduct the experiment to allow us to compare the advantages and disadvantages of the common technologies for conductor forming (table 1).

Productivity: for “V”-shape conductors – it is lowest in comparison with all others, because all production steps are carried out successively in time (score – 1 point). Repeatability of the conductor crown: “S”-shape conductors have high repeatability (score – 3 points), whereas “P”-shape conductors have low repeatability (score – 2 points), because the conductor crown is freely formed during the time twisting process.

Optimal marker dimensions of the conductor crown: only the “S”-shape section allows for fine adjustments of the crown dimensions (score 3 points).

Airflow: minimal airflow in the basket can be observed in the case of “P”-shape section, because the slope of the crown shoulders cannot be controlled (score – 1 negative point). This problem can be solved by applying additional shifting in the direction of the Z-axis.

Practice shows that only “S”-shape can fully optimize the gauged dimensions of the basket and ensure minimal basket width (score – 3 points).

Minimal crown height can be achieved using the “V”-shape conductor (score - 3 points).

“P”-shape has a high risk of shorting in the crown shoulders (score – 1 negative point).

“V”-shape does not allow for any corrections in the crown shape (score – 1 negative point).

“V”-shape is not suitable for varnished conductors because it can easily damage the insulation (score – 1 negative point).

The total scores show the benefits of “S”-shape conductor in comparison with all other section types – 27 points (table 1).

Tabl. 1.

	“V”- SHAPE	“P”-SHAPE “U”- SHAPE	“S”- SHAPE
1. Productivity	+	+++	+++
2. Crown shape consistency	+++	++	+++
3. Optimum overall crown dimensions	++	+	+++
4. Maximum air flow through the winding	+++	-	+++
5. Minimum winding width	++	+	+++
6. Minimum crown height	+++	++	+++
7. Risk of short in the winding	++	-	+++
8. Crown shape adjustment	-	++	+++
9. Enameled wire	-	+++	+++
BALANCE	14 +	12 +	27 +

CONCLUSION

The experimental results show that the proposed “S”-shape has significant benefits when compared to all other conductor types.

We can conclude that the new highly effective “S”-shape conductor and the technology used for its forming can find applications not only in the hybrid, but also in the conventional engines for the automotive industry.

THEORY OF PRECISION MECHANIC PANEL SCREWS

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Abstract: Constructions belonging to precision mechanics need to be suitable for the functional conditions. The product has to perform the task reliably in its perfection in the prescribed limit of error. At the same time this aim must be reached not at any price but economically, well manufactured and in easily marketable realization. A new construction usually becomes out of date in three-four years morally deteriorates even though if it is useful in the aspect of working. This is the explanation of the fact that the standardization and the normalization in precision mechanics play a great role. During the latest decades the polymer pieces have been appearing in the different areas of the industry. Nowadays polymer and composite materials are used not only as covering but as the material of concrete mechanical elements as they have a wide workable and usability spectrum. The pieces made of polymer materials in the telecommunication industry take an honoured place. It is because they are good insulators they are light and cheaper than the metal materials. In the European Union it is a regulation that the most of the pieces must be recyclable. It means not only the reuse of the raw material but the recycle of the pieces. So the new and economical types of connection in the polymer pieces have appeared, and their examination have become important, too. The self-tapping screwed joint is suitable, too, the economical point of view and for the recycle, as it does not need any more operations after the removal of the screw.

Keywords: SELF-TAPPING SCREWS, VOLUTION, THREAD CUTTING, TAPPING, BOLT PRELOAD

1. Introduction

During the latest times masters made an effort on optimization of geometry of the screw depending on the material of the tube. The several finite element models mean great help not only with the investigation of the physical processes during the creation of the screwed joint but the examination of mechanic behavior of the bolt joint because it makes possible the forecast the influence of the parameters on the physical process.

If there is self-tapping screwed joint there is no need for threaded boring made in advance, as the screw makes the female tread during the process of the screwing in. So the self-tapping screwed joint is the particularly economical form of the threaded bonds. The user is not often able to control the quality of the threaded bonds because of the high speed of the electrical screwdrivers. Screwdrivers used wide for the steering of the screwing in process use a simple built in torque limiter, which breaks the screwing in process if the torque departs from the posed value. The automatic control of the screwing in process is very important because of the great number of the threaded connections used in the practice. If we make a comparison between the torque-displacement graph measured during the screwing in process and the theoretic torque-angle of rotation characteristic of the threaded bonds then the producing of screwed-joints become watched over. We can reach it by the way of making comparison between the torque-distance graph on the screwing bond being shaping and the graph of a well-produced screwing connection. Insertion of measured in advance curves causes significant surplus work if we use more different screwing bonds.

Table 1: Possibilities of fixing polymer elements with screwed joint

Screwed joints	Without inserts			With metal insert
Nut thread	made in advance	screwed joint		
Volution		thread cutter	thread tapper/driller	
Screw type	metric screw	screw thread with cutter edge	tapping screw (for metal or wood)	screw with metric thread
Adaption	sometimes, complicated, expensive	brittle materials: fiber-reinforced thermo-plastic polymers	tough materials: thermo-plastic polymers	often dismantling bonds, foamed polymers

Tab. 1 shows the screwing bond solutions of the fixing of polymer pieces.

We can distinguish two kinds of screws: forming and cutting threads. Forming threads are used as tinners screws or wooden screws. Producing this connection remarkable stress come into being in the tube, so while choosing the material it is very important that the material will be able to enlarge to avoid the polymer geol. While making the cutting threads so remarkable stress do not generate into the tube because the screw produces the nut thread by shaping.

The screws used in practice are shown in Tab. 2 and the process of producing the screw-joints are displayed on Fig. 1.

Table 2: Types of self-tapping screw (used for polymers)

Name	Screw type	Properties	Application	Schematic
AB	Thread forming	Do not contain cutting slots Do not produce chips 60° thread angle	Materials with a modulus less than 2760MPa	
B	Thread forming	Large root diameter, finer thread pitch	Designed for wood and metal, but also used for thermoplastics	
BF	Thread cutting	Wide thread spacing can suffer clogging of the cutting slots when working with softer materials	Materials with a modulus greater than 2760MPa	
BP	Thread forming	Same dimensions as B, but has a cone point for use where holes are slightly misaligned	Materials with a modulus less than 2760MPa	
BT	Thread cutting	Most common Wide thread spacing	Materials with a modulus greater than 2760MPa	
D	Thread cutting	Good for field replacement	General use	

F	Thread cutting	Blunt tapered point Multi-cutting edges Chip cavities	Both metals and plastics	
T	Thread cutting	Fine thread spacing to reduce granulation	High modulus (greater than 7000MPa) glass reinforced materials	
Hi-Lo	Thread forming / thread cutting	Double lead screw -high thread 30° - low thread 60°	Designed for thermoplastics Excellent pull-out due to the increased contact with plastic between the high threads	
Trilobe	Thread forming	Triangular spaced threads -single lead - double lead Thread angles 45-48°	Increased resistance to loosening, ideal for vibration applications	

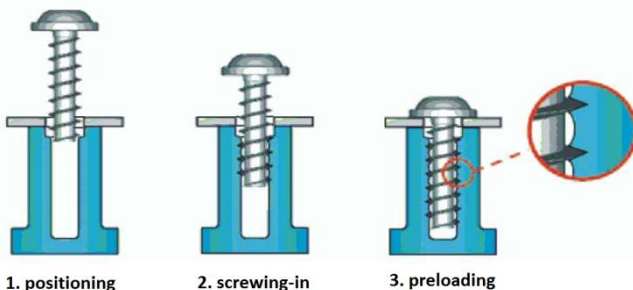


Fig. 1 Main steps of making self-tapping screw bonds

Torque on the screw during the process of screwing in devotes to the shaping of the threads and friction resistance between the screw threads and the tube. During the screwing in we have to reckon on heat generation because of local, plastic deformations and the sliding friction. Added difficulty that the force occurred during the process changes because of the behavior of the polymer (creeping, stress relaxation). The determinate elements on the quality of self-tapping screwed joint are shown in Fig. 2.

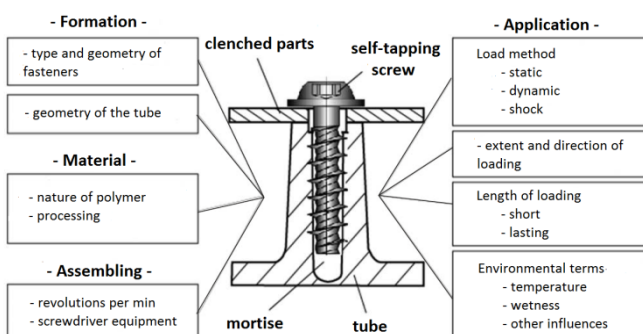


Fig. 2 Factors determining quality of self-tapping screw bonds

Screwing bonds used for the fixing of thermoplastic polymer elements make two types:

- The first group contains those bonds where the piece is fixed between the screw-head and the front of the tube. So the polymer piece will be exposed to a nearly permanent that is independent from time-change. (the synchronous stress-strain curve can be displayed by a vertical line which shows the influence of the time during the process from the pre-stress of the screw)

- In the other group an added loading influences on the bonds. A nearly permanent stress is in the polymer device.

It is worth to mention that the geometry of self-tapping screwing joints used nowadays has been developed by a continuous development work, which can be seen on the Fig. 3.

2. Directives for selection and dimensioning

Research result of this essay applies exclusively the pairing of self-tapping steel screw and polymer tube.

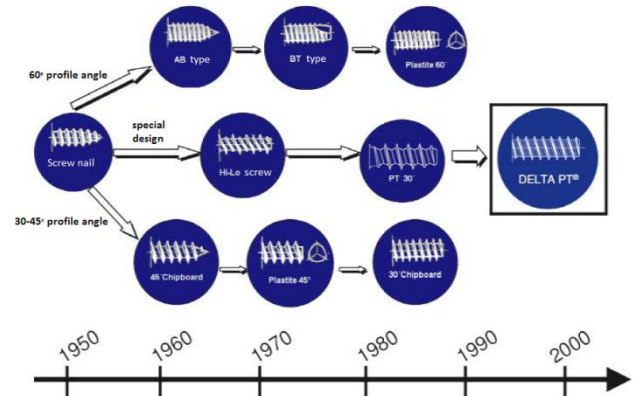


Fig. 3 Changes in the geometry of the self-tapping screws

2.1 Forming the screw

The diameter of self-tapping screws used in thermoplastic polymer devices changes 1.4-10mm. The threads are less profile angled (45°, 40° or 30°) in the screws so as to be able to reduce the radial stress in the polymer tube. The thread ditches are also deeper than for example in the case of metric threads. The used thread profiles are changed by the producers.

An optimal thread has to be adequate to the following criteria:

- the radical shape-change of the tube must be small
- it has to demand small screwing-in torque
- the size of the connected hob surface must be as big as it can be
- a narrow tolerance field has to be particular to it

The cutting edge working with brittle materials has got a big importance in the lightening of screwing-in. Collar-screws are often made to minimize the stresses on the beddings of screw-heads. The Fig. 4 also displays a screw like this.

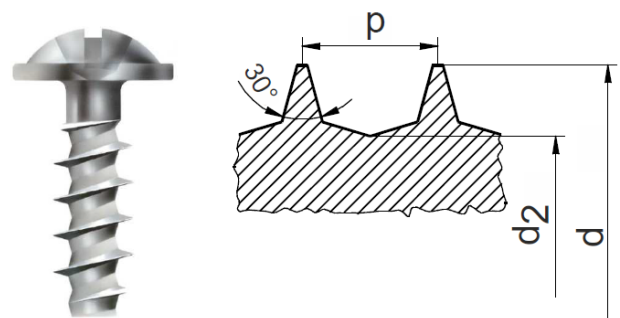


Fig. 4 PT type screw

On it we can see the threads, too. Next to the 30° profile screw we can also see, that the geometry of screw bar is different from the traditional metric screws. This is made so that the ditch can be filled by the polymer material. By the screws used nowadays we can reach suitable pre-stress even after numerous unbinds a pre-forces. Screws used with polymer tubes are characterized by the followings:

- smaller inner diameter, bigger thread height with the metric thread
- higher thread lifting
- 30° profile angle

2.2 Forming of tube

Fig. 5 shows the scheme of a polymer tube. On the polymer device the fixing points are made as a cylindrical inner core (tube). The tube is lift up with $0,5-1^\circ$ taper to make the device to be capable to let the screw out of itself - an inner so called relief bore is made on the top surface of the tube - often with a bigger diameter (d_s). This bore lightens the fitting of the screw in the bore ($d_s > d$) and relieves the top surface from the loadings added by the threads. The best forming of the tube is the condition of achieving the maximal pre-stress force.

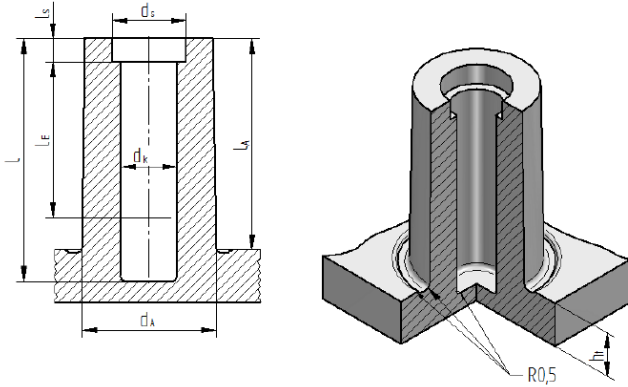


Fig. 5 Schematic illustration of the polymer tube

If the length of the screwing is longer the bearing of burdens of the screwing bond is growing. The solidity of the screw means the top limit of bearing of burdens. On the contrary the metal screw – metal female the metal screw – the polymer tube's each thread takes part in the load delivering, which is the result of elastic modulus of the two materials.

2.3 Pulling torque

Fig. 6 displays the torque on the screw-head during the making the screwing joint taken as a function of the turning round the longitudinal axis of the angle.

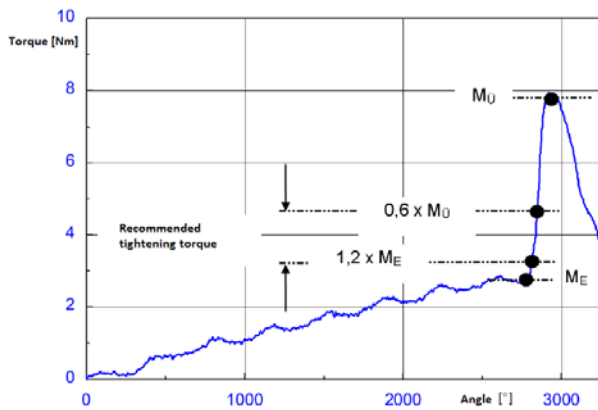


Fig. 6 Torque effect on self-tapping screw depend on the angle

The bigger the speed of the screwing in the smaller the torque (M_E). This influence is stronger with amorphous thermoplastic polymers than with the partly crystalline materials. This is explainable by the friction heat and as a result of it with the material softening. It is also foreseeable that the greater the biasing force the bigger the load of the polymer tube will be. The higher biasing force demands the higher relaxation at the same time. Pulling torque is

$$(1) M_A = M_E + k(M_U - M_E)$$

where the k 's value is 0.2-0.5. By Erhard the value of k depends on the parameters of the screwing machines. Screwing in torque (M_E) is that torque, which we see when the screw-head is only set on the surface of the polymer tube, but there is not pre-stress yet. Two things make the screwing in torque forming threads and the friction. (M_U) is that torque which is used then when the bond deteriorates.

2.4 Biasing force

We can avoid the overloading and the too strong biasing force relaxation if we choose the value of the pre-stress very carefully. It is not effective to use too big biasing force during making screw bonds. Experiments gave evidence that with a bigger screwing-in speed (500-1000 turning/min) we can achieve bigger biasing force with give sized pulling torque than in case of smaller speed. We can normally estimate the biasing force in the joint (F_V), but the accurate value of it is must be measured. We suppose that the linear connection is between the biasing force and pulling torque. The biasing force is:

$$(2) F_V = \frac{4F_A \cdot k(M_U - M_E)}{4(M_U - M_E) + F_A \cdot \mu_F \cdot (D + d_s)}$$

if we consider the friction between the surfaces. If we take a steal-polymer sliding couple the friction fact is 0.2-0.3.

The biasing force in time continuously decreases because of the polymer material's time and temperature additive behavior. Experiments demonstrate that in average working circumstances there is no need for a big biasing force decreases in bigger degree taken as a function of time than a smaller biasing force.

3. Results of discussion

We have a detailed literature connected to the screwing joint. Experts have investigated various aspects of screwing bonds for example the geometry of screw, the stability of the joint, the parameter of setting-up, the possibility of automation of setting-up, the forms of crash. These parameters have been investigated by theoretical ways and by models. On the basics of these theoretical investigations they have created connections based orientation on adoption, which present a basis for choosing the material or the measurement of the bolted joint. Nowadays experts often describe the behavior of the joint or the different work phases by numerical models.

The screwing machines in most cases have got torque limit, which undo at a given pull torque and at the same time it ensures the same pull torque to each screwing joint. During the latest decades higher and higher demand emerged on the automation of the screwdriving process, which have developed to a reliant area of research. The aim of these researches is the development of the automatic controlling and supervisory strategies in support of increase of production. The aim is to make a model, which can determine that in the case of given pull torque how big is the axial moving away of the screw.

Material of the plate used for producing the female was ABS (acrylic-butadiene-styrene) and PC (polycarbonate). Using the model we need data of the geometry of screwing joint (parts we need to fix), the characteristics of the material, friction facts, the diameter of the pilot hole and the geometric measures of the screw. On the Fig. 8 we can see the theoretical torque-turning away curve belonging to the PC female. The numbers on the table show the characteristic linear phases of the graph.

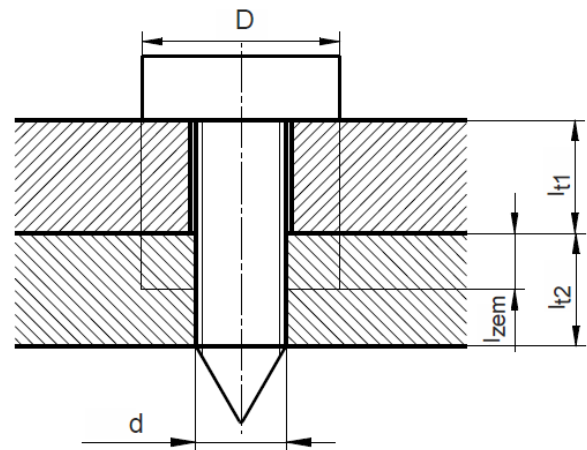


Fig. 7 Model of self-tapping screw joint with abutment screw head

Phase 1: The cone-shaped end of the screw is touching the pilot hole of the female (Fig. 9 T0). Then the screw is going into the female by the time when its tang touches the pilot-hole (Fig. 9 TE). The first step of the screwing-in process, the screw is snatching at the female. The torque which influences on the screw is devoting to the overcoming of the resistance caused by the making thread and the friction.

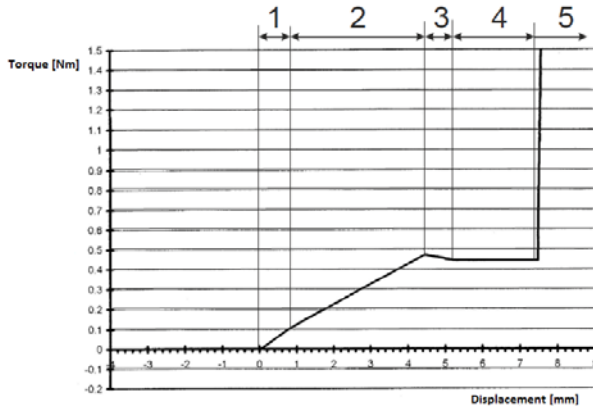


Fig. 8 Theoretical displacement-torque graph of polycarbonate female ($l_{t1}=0\text{mm}$, $l_2=3\text{mm}$, $E=2.3\text{GPa}$, $p=1.1\text{mm}$, $D=5.03\text{mm}$, $d=2.87\text{mm}$)

Phase 2: The screw is continuously going in the pilot-hole (Fig. 9 TP). The torque on the screw is now devoting to the overcoming of the resistance of the thread making by the cutting edge and the friction between the screw and the female thread. The thread in the female is growing and it causes more and more friction resistance.

Phase 3: The cone-shaped end of the screw is turning up at the end of the pilot-hole (Fig. 9 TB), then it is emerging step by step till the cylindrical part of the screw is at the end of the pilot hole. In this phase there is thread-making and friction yet. The ready thread made in the female at the end of this phase achieves its final length.

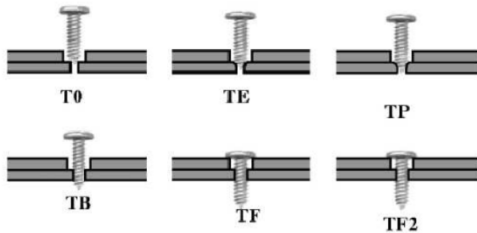


Fig. 9 Phases of making self-tapping screw joint

Phase 4: The cylindrical part of the screw is turning up at the end of the in and out pilot-hole (Fig. 9 TF), then it is coming out. In this phase there is no thread-making, the torque on the screw is totally devoted to overcoming the resistance of the friction on the threads. The torque is the same, because the length of the thread in this phase does not change, so the friction resistance remains the same, too.

Phase 5: The head of the screw lies on the piece (Fig. 9 TF-TF2). The torque on the screw is devoted to the overcoming of the resistance friction on the surface of the screw-head.

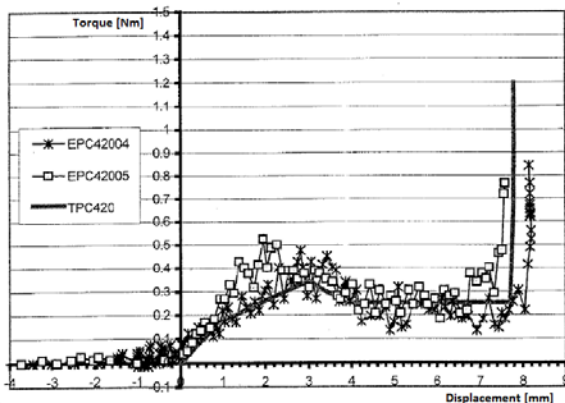


Fig. 10 Theoretical (unmarked graph) and measured (graphs with symbol) torque-displacement graphs in case of polycarbonate female

The different conditions of the 5 phases are well seen on the Fig. 9. And at last the comparison of the estimated and the measure torque-moving-away graph is on Fig. 10. The results show good conformities, which support the usefulness of the model.

4. Conclusion

Seneviratne and his fellows report graphs given with survey data torque-moving away. These measures prove the truth of the presence of the 5 phases in the theoretical models.

Althoefer and his fellows presented a new method for the supervision of the screwing joints. The elaborated theoretical built on the torque-moving away graph artificial neural network can distinguish the well or badly made screwed joints. By the authors the torque-displacement graph using the model depends on the geometric and mechanic characters of the fixing pieces and the screw that join them each other. The curve depends on the strength modulus, the friction fact, the solidity of the materials, the thickness of the pieces which are joined each other, the diameter of the pilot-hole and the measures of the screw. The graphs connected to the different pilot-hole diameter can be seen on Fig. 11. The comparison of the theoretical model and the torque-displacement graphs definite by measure is shown on the Fig. 12.

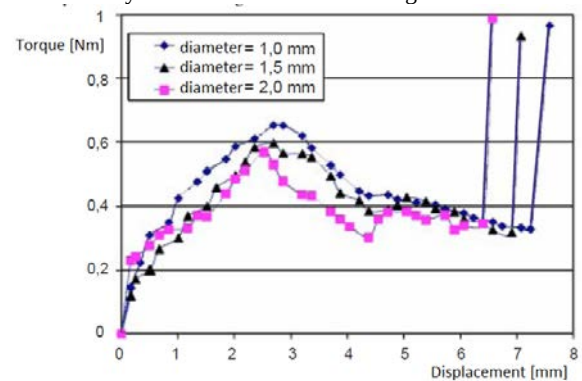


Fig. 11 Torque-displacement graphs for different pilot-hole diameters

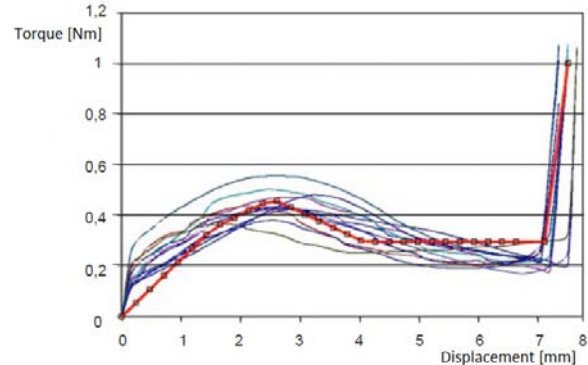


Fig. 12 Comparison of measured (thin lines) and calculated (thick lines) torque-displacement graphs

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OPTIMISING TECHNOLOGY FOR PRODUCTION OF HIGH FREQUENCY WELDED PIPES MADE OF X60 STEEL

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ABSTRACT: In this research work the Welding technology that implements high frequency contact welding process (HFCW), is presented as a method for automatic steel pipes production. The pipes produced by the HFCW are meant to be used for natural gas transportation. X60M steel coils with thickness of 6.5 mm are used as a raw material. The outer pipe diameter is $\varnothing 508$ and the length is 12000 mm. The preliminary welding procedure is based on the personal experience, technical knowledge and the expert judgment. A special attention has been paid to the most important welding parameters such as: current frequency, voltage and welding speed. After the welding was finished, an additional heat treatment (normalization) of the welded pipes (joints) was performed at the temperature of 900°C.

In order to justify the proposed welding technology a complex analysis is applied. The first analysis was the chemical analysis of base metal. The next was the nondestructive testing consisted of a visual inspection, and ultrasound testing of the pipes. The destructive testing was consisted of tensile testing, bend testing and the hardness measurement. Besides the analysis of the micro and macro structure of the welded joints, a metallographic analysis is performed as well. Moreover a hydrostatic test and a flattening test were performed as an integral part of the entire testing process. The test results confirmed that the pipes produced with the predicted welding parameters completely fulfilled standard requirements. The manufacturing process for the pipes for natural gas transportation is ready to begin.

KEYWORDS: HIGH FREQUENCY, WELDING, CONTACTORS, X60 STEEL, PIPES, MICROSTRUCTURE

1. Introduction

Over the past few decades' severe demand were placed to the pipes manufacturer with respect to the development and processing of the material for the pipelines. Generally longitudinally welded steel pipes are used for gas and oil transportation because of the high safety and as an economical method of manufacturing. Because of the conditions where the pipes are used such as high ground pressure, they must possess high strength and toughness [1].

More than eight millions tons of steel pipe are produced in the world every year. Most of them are produced from standard steel grades. The bigger challenge for the pipe producers is the manufacturing the pipes from X60, X70 and X80 which on the other hand requires special efforts and measures [2,3].

High frequency tube welding is one of the most forgiving industrial processes nowadays, and it is possible to produce acceptable tubing for most purposes. High frequency welding is a form of electrical resistance welding (ERW). A voltage is applied (HF contact) or induced (HF induction) across the edges of the open tube just prior to the point of closure. This voltage causes a current to flow along the edges to the point where they meet, causing rapid heating of the metal. Pressure is applied by the weld rolls, which forces the heated metal into contact, forming a hot diffusion bond. The pressure also forces the molten metal and any impurities out of the weldment [4]. The only real difference between high frequency contact and induction welding is that with contact welding, the voltage is applied directly to the strip edges by means of sliding contacts, whereas in the case of induction welding, the voltage is induced by the magnetic flux surrounding the coil. The reason for using a higher frequency is that in the case of induction welding, it is desirable to keep the size of the coil reasonably small. The higher the frequency, the less flux is required. The higher frequencies also affect the behavior of the current that flows in the "vee". As the frequency is increased, the current tends to concentrate closer and closer to the edges of the strip. This is partly due to the "skin effect" which causes the current to flow on the surface of conductors at high frequency, and partly due to the "proximity effect" which causes the current on the adjacent conductors to concentrate at the adjacent surfaces. Both of these effects are caused by distortion or interaction between the magnetic fields associated with the current flow [5,6]. Frequencies used for tube welding are in the range between roughly 100 kHz to 800 kHz, with lower frequencies being used for large, heavy wall tube, and the upper range being used for small, thin walled products, especially those using nonferrous material. The current that flows along the edges of the strip to the apex of the "vee" heats the strip to the welding temperature. The current will also tend to flow around the inside circumference of the open tube. This heats the entire tube, and does not contribute to the

welding process. Thermo-mechanical control process (TMCP) is one of microstructural control techniques, combining controlled rolling and controlled cooling, to obtain excellent properties of the steel plates, such as high strength, excellent toughness and weld ability [7]. The controlled rolling and the controlled cooling are to satisfy the high strength and high toughness requirements, which could only be achieved conventionally by off-line heat treatment. Located close to the mill, the practical operation type also gives an opportunity for higher productivity and shorter production time that are always demanded in industrial products [8].

The large-diameter steel pipes are produced by high frequency seam-welding followed by induction-assisted heat treatment. The pipes for oil and the gas transmission are made from low-alloy steels designated X60, X65, X70 etc. depending on the strength and toughness required and the intended application. One of the methods of fabricating pipes from these materials is by high-frequency contact seam-welding [9-11].

The toughness of the line pipes is paramount in their suitability for application. The joint resulting from the contact welding is quite narrow, with a central 2 mm wide region, but it also represents a source of weakness. That is why the welding process is immediately followed by induction heat treatment. The intention of the latter is to refine the microstructure by reutilization at a lower temperature [12].

One of the TMCR are the alform series i.e. thermo mechanically rolled, weld-able and bendable fine grain structural steels. The plates made of these steels combine the good toughness properties of the thermo-mechanically rolled fine-grain steels according to EN 10025-4 with the excellent cold forming properties of the cold forming steels according to EN 10149-2. The alloying concept provides very low carbon contents and low carbon equivalents, which aims to a very good weld-ability. In particular, the high-strength grades provide special advantages in areas, where weight the savings are of a great importance. Although the heat-treatment improves the toughness of the welded region, the increase is not as large as might be expected from the reduction in the scale of the microstructure [13].

Therefore the basic idea of this research work is to verify the proposed welding technology (using HFCW process) for production of the steel pipes (for natural gas transportation) made of micro alloyed steel X60M.

2. Material and experimental

Raw material

The raw material used (steel coils) for the pipes production was micro alloyed steel with designation X60M PSL2. The designation of steel is according to API 5, 45th Edition/ISO3183. Explanation of

the designation is the following: X means steel for pipe production according API. Digits 60 ksi concern to yield strength of the steel i.e. to 420 MPa. M means that steel is produced by thermo mechanical rolling process. PSL refers to the product specification level where PSL2 provides a more extensive chemical composition complete with a mandatory minimum. The chemical composition and mechanical properties of the raw material are given in the tables 1-3.

Table 1 Chemical composition of the raw material measured and according API standard requirement

El.%	Measured	Standard
C	0.074	0.12
Si	0.184	0.45
Mn	1.17	0.16
P	0.012	0.025
S	0.004	0.015
Cu	0.0233	
Cr	0.024	
N	0.007	
V	0.0403	
Nb	0.0357	
Ti	0.0111	

Table 2 Mechanical properties of the raw material and weldment

Tensile test			
	Rp0.2	Rm	A%
BM	527	588	32.7
WM	530	584	-

Table 3 Impact toughness of raw material weld metal and HAZ

Charpy V-notch, 0°C				
	1	2	3	Av
BM	210	208	207	205
WM	190	180	165	179
HAZ	203	200	185	196

Table 4 Typical parameters used for welding of HFRW

Welding parameters	Values
Welding speed (m/min)	22
Voltage (kV)	14,3
Current intensity (A)	23-24
Current Frequency Hz	400
Generator power kW	600
Voltage index	57-58

The raw material used for the steel pipe production was in a form of coils. The coils had to be exact width-ness in order to allow the proposed diameter of $\phi 508 \times 6.5$ to be achieved. The coils are delivered to the starting point of the technological line (Figure 3a) from the store. (Figure 1). Then the coils are moved (Figure 2a) to the technological production line by cranes. The round shape of the pipe is achieved with the rolling mill technology where the rolled strips are formed into a circular cross section by funnel arrangements of rolls. The size of the rolls is chosen as per the diameter of the pipe and the thickness of the material. (Figure 2b) The pipes are then welded implementing the electro resistance contact welding process. (Figure 2c.) The beginning of the pipe welding process and the V-wedging are given in (Figure 3b). The parameters of the welding wedge play a very important role in terms of the quality of the welded joint. The softened and melted edges of the pipes are connected by the pressure of the squeezing rolls (Figure 3). The excesses material and impurities are squeezed

out on the inner and the outer pipe surface. (Figure 4). The removal of the material (slag) is performed with special knives located on the inner and outer side of the pipe. After the excess material is taken off, the welded pipes are cut in the length of 12 meters. A circular mechanical saw is used for cutting the pipes. (Presented in Figure 2d).

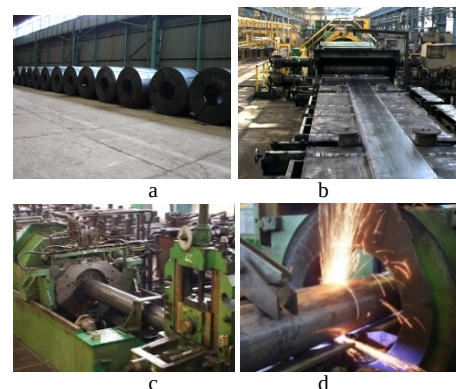


Figure 1(a-d) Production of steel pipe from the raw material API X60 steel
a. Store for raw material b. involving raw material in the production line c. welded pipe d. cutting of the pipe

As is shown in the figure 5, the welded pipes are then additionally exposed to a heat treatment (normalization) in order to improve the properties of the welded joints (relaxation of stress, microstructure....). Normalization is performed at the temperature of 900 °C by 6 inductors. The width of normalized layer is about 20 mm.

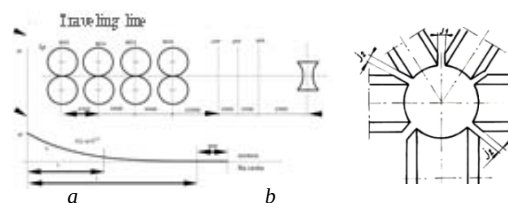


Figure 2 a Technological line for contact welded pipes production b. final forming of pipe before welding b. formed pipe before HFCW

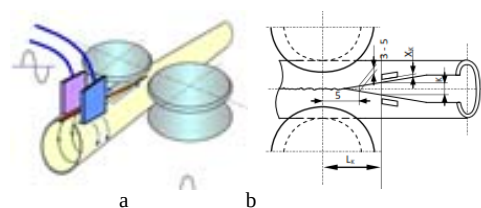


Figure 3 Electro resistance contact welding of pipes a. Contactors b. Welding start point and V-wedge

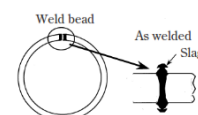


Figure 4 Pipe with excess material (slag)

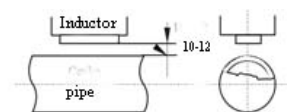


Figure 5 Induction heating of weld with 6 inductors

Evaluation of the preliminary welded pipes

Many complex testing and evaluations were performed in order to confirm quality of the welded joints. A welded pipe was subject to

hydrostatic testing under the pressure of 10 bar for period of 10s. There was no leakage after the testing. For the flattening test, two segments of 200 mm were cut and tested under pressure. Position of the welds during the testing is shown at (Figure 6). Applying the pressure to the segment last until the opposite sides of the pipe touch each other. No defect like cracks were detected during testing.

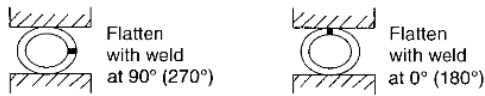


Figure 6 Flattening test of pipe segments

A visual inspection was performed as well in order to detect surface defects or the geometrical misalignment of the pipes edges. The testing was performed as per the BS EN ISO 17637:2011, and no any defect were detected.

From the tested pipe specimens were cut for mechanical testing. Transferral tensile testing of the welds was performed according to the EN ISO 4136. Another three specimens were prepared for the perpendicular tensile testing of the welded joint. The result are given in (Table 2). During the all tests the breakage of specimens was out of the welded joint. The obtained testing values are almost identical with the base metal properties.

The bend testing was performed according to the EN ISO 5173:2010+A1:2011 standards. The results of testing are given in the (Table 5). As a result the all tested probes fulfilled regulation requirement.

Table 5 Results of bend testing of welded joint

Nr.	Type of test	Dimension of specimen	Bending angle	Results
1	TFB	6.5x30	180 ⁰	good
2	TFB	6.5x30	180 ⁰	good
3	TFB	6.5x30	180 ⁰	good
4	TRB	6.5x30	180 ⁰	good
5	TRB	6.5x30	180 ⁰	good
6	TRB	6.5x30	180 ⁰	good
7	LRB	6.5x30	180 ⁰	good
8	LFB	6.5x30	180 ⁰	good
9	SB	6.5x6.5	180 ⁰	good
10	SB	6.5x6.5	180 ⁰	good

Meaning of the abbreviations is the following:

TFB – transferral face bend, TRB – transferral root bend, LFB – longitudinal face bend, LRB – longitudinal root bend, SB – side bend.

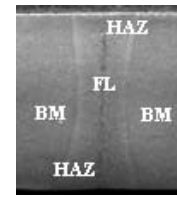
The impact toughness testing was performed according to the EN ISO 9016:2012 and EN ISO 148-1:2010. Besides the base metal, the weld metal and HAZ were tested too. The results from testing are given in the (Table 3). Impact testing was performed at temperature of 0°C. It has to be point out that approximation of obtained results was performed. The reason is that nonstandard Charpy specimen were used because the thickness of the pipe was 6.5 mm, which is nonstandard. The charpy specimens has to be with thickness of 10 mm.

Macrostructure of HF welded joint is presented in the (Figure 7). On the same specimen was performed hardness testing according to the EN ISO 9015-1. The hardness testing was performed across the line parallel with the welded surface. Vickers method (HV10) for hardness measurement was implemented. The highest hardness was noticed in the weld metal and the lowest in the base metal. As per our expectations, the normalization lower the hardness of the welded joint.

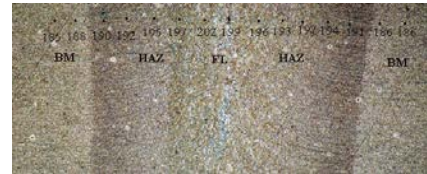
From the macro photo can be clearly seen characteristic area in the welded joint. In the middle of the joint is the fusion line after that is HAZ and the base metal which does not suffer from welding heat input. No allowed welding defects are detected. It can be seen that

the flux i.e excess material is formed during the welding. As can be seen from the macro photo squeezed metal is cut (removed).

Typical welding defects which can be formed during HF electric welding like cold laps, entrapped inclusions, cold weld, open weld, cross weld, cracks in the weld porosity cracks in the welding zone.



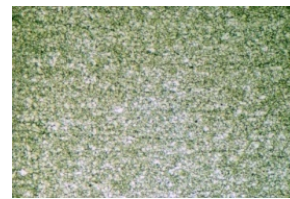
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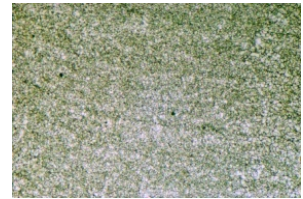
b

Figure 7(a and b) Macro photos of welded joints

Microstructure of the base metal is presented in the figure a. It is fine grain ferritic-bainitic. The fine grain HAZ zone can be seen in the (Figure 8b). Both microstructures are very similar. Deformation of the microstructure as result of the pressure of the forming rolls can be noticed in (Figure 8b). The fusion line co- called ferritic line is given in the (Figure c), where can be seen that the microstructure is coarser. As a dominant microconstituent in this area is block ferrite.



a



b



c

Figure 8 (a-c) Microstructure of the welded joint

3. Discussion

The experimental welding was performed as per the previously set parameters, based on the technical knowledge and the experience from the similar application. After finishing the experimental welding, an additional heat treatment (normalization) is performed on the welds of about 20 mm wide.

A variety of measures are taken to ensure that the finished steel pipe meets specifications. In order to check the properties of the pipes very complex mechanical, chemical microstructural and technological testing and evaluation were performed. (Mainly according to the API 5L standard). The quality of the raw material was checked before the welding starts and was done exactly as the regulation required. The visual inspection showed that there were no geometrical defects of the pipe or of surface welded area. The automatic ultrasonic testing was done directly in the production line, and didn't detect any volumetric welding defects. The hydrostatic test of the pipe at the pressure of 10 bar/10s didn't show leakage in the weld or any visible cracks. The flattening test of the pipe segment, 200 mm wide didn't reveal any defects in the welded joint as well. The hardness test showed that the max hardness is in the WM, slightly better hardness was measured in the HAZ and

normally base material showed the lowest values. On the contrary the measured impact toughness values were quite opposite so the best values has the BM than the HAZ and the lowest value has the fusion line. However all the values are much higher than the current standards require. The tensile testing showed that the results are similar to those of the raw material, no matter the breakage of the probe was always in the base metal.

4. Conclusion

This report shows that the welding technology implementing high frequency contact welding process (HFCW), is a suitable method for automatic steel pipes production. During the research process a special attention was given to the key parameters which affect the quality of the final product. Numerous tests and valuation of the results was conducted in order to define the key parameters and their values.

The results showed that X60M pipes manufactured with high frequency contact welding process (HFCW) meet the requirements of the API 5L standards. Therefore the manufacturing of the pipes is ready to begin.

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ONTOLOGY-BASED DATA ACCESS AND MODEL TRANSFORMATIONS FOR ENTERPRISE INTEROPERABILITY

ОНТОЛОГИЧНО БАЗИРАНИ ДОСТЪП ДО ДАННИ И ТРАНСФОРМАЦИЯ НА МОДЕЛИ ЗА ОПЕРАТИВНА СЪВМЕСТИМОСТ НА ПРЕДПРИЯТИЯТА

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Abstract: Enterprise interoperability is the ability of disparate and diverse information and control systems to work together efficiently towards mutually beneficial common goals. Some of the main tasks for achieving enterprise interoperability are connected with data and knowledge access and sharing and by use of model transformation. In this paper are presented and discussed several model transformations between different Technological Spaces: RDB, UML and OWL in order to enable ontology-based access to different models of enterprise data. At the core of the approach is the usage of reference models, based on the standard for enterprise integration ISO/IEC 62264.

Keywords: ONTOLOGY, INFORMATION, INTEGRATION, OWL, UML

1. Introduction

Information access and data retrieval from distributed heterogeneous data sources are important, but difficult tasks in the modern industrial enterprises. Some of the possibilities in this direction are standard frameworks and data models, based on the best practices, which separate the information and knowledge from specific commercial products and methods of implementation. In this context, the ISO/IEC 62264 standard [1] (former ANSI/ISA S95) provides common terminology and consistent reference models that represent the best practices for integration, applicable the entire life cycle of enterprises. The reference models, provided by the standard, are built on a high level of abstraction, independent from the specific industry and might be used as a basis for development of any enterprise systems as well as for standard compliant interfaces between the legacy systems.

A major trend in enterprise systems over the last decade has shifted from the traditional programming of imperative code to the creation of rich, interchangeable representations of information and knowledge [2]. Two significant paradigms (also known as Technological Spaces (TS) in [3]) have emerged in recent years to support this activity, each with its own terminology, standards bodies, research communities and flagship language: Model Driven Development (MDD) evolved primarily in the software engineering community and has the OMG's Unified Modeling Language (UML) [4] as its flagship language. The other is the so-called ontology engineering paradigm that places ontologies at the center of the development process. Ontology engineering primarily evolved from the artificial intelligence community and has the W3C's Web Ontology Language (OWL) [5] as its flagship language. Several publications discuss the relation of UML and OWL in general and the transformations of UML class diagrams into OWL ontologies. In [6] the authors investigate whether and how conceptual models written in UML can be automatically transformed into models written in OWL 2. Differences and similarities of various model elements in static UML data models and OWL 2 ontologies are shown and a transformation for similar elements is provided, indicating that data models written in UML can be represented as OWL ontologies quite well. But the different extent of the meta-models clearly suggests that OWL provides much more complex means of modelling already, so the transformation of general ontologies in UML data models is not always possible [6]. The transformation into an OWL 2 ontology allowed to check the consistency of the UML models and the data [6]. In [7] we discussed the two different approaches for a transformation between

UML and OWL 2: XML-based transformations and transformations built upon meta-models using transformation languages (ATL, QVT-R). In this paper we introduced transformation rules in order to provide semantic descriptions of the object models proposed in ISO/IEC62264 standard, which use only few UML modeling elements. The suggested rules are built upon the meta-models of the UML class diagrams and the language OWL 2. The paper [8] also investigates providing semantic descriptions for particular enterprise models with limited UML elements, in order to promote system interoperability, enterprise information integration and information retrieval.

Relational databases are considered as one of the most popular storage solutions for various kinds of data. But according to Honeywell Process Solutions [9] relational data models can be very complex, difficult to report, inflexible. The variety of DBMS implies synchronization across data sources. Using data warehouses duplicates data and is not suitable for real time. Contrariwise, semantic data models are easier to understand, easier to report, flexible and can merge any data without replication; they are able to perform true federation: support structural change and system discovery across data sources [9]. The problem of bridging the gap between relational databases and semantic web ontologies has attracted the interest, and has been recognized as a key factor in generating huge amounts of data for Semantic Web applications based on standards RDF, OWL, SPARQL [10]. Transforming structured data sources into universal description model, as RDF aspires to be, enables seamless integration of their contents with information already represented in RDF [10]. A lot of methods and tools, classified in Knowledge Extraction Tools Survey Ontology (KETSO) are analyzed in [11]. Since data management according to the relational data model is still an order of magnitude faster than RDF data management and RDBs are the dominant data storage solution for enterprise data, relational data management will be prevalent in the next years.

Compared with relational databases, object data models are easier to understand and also can merge measurement's data [9]. The Object-Relational Mapping (ORM) enables transformations between object models and relational databases. For application development it is easy to start from creating the model by using the UML Class Diagram to generate the executable persistence layer for the model. By Model Driven Development (MDD) approach using models at different levels of abstraction and applying model transformation to code generation, it is useful also and to modify the Entity Relational (ER) model, which comes from reverse engineering of an existing database, transform into object model

and generate persistence layer. Mutual software tools automate the Object-Relational Mapping enabling the MDD of different software systems.

The aim of the paper is to present and discuss several model transformations between the three Technological Spaces (TS): the DBMS TS (with a representative RDB), the Model Driven Architecture TS (with a representative UML) and the Ontology Engineering TS (with a representative OWL) in order to enable ontology-based access to different models of enterprise data. The basis of the approach is the usage of a standard for information integration during all phases of the life cycle of systems.

The paper is organized in 5 parts. In part 2 a short overview of the basic technologies and tools used in the approach is given. The third part of the paper discusses the approach for ontology based data access and information retrieval. In Part 4 the applicability of the suggested approach is illustrated. Finally some conclusions are made.

2. Basic technologies and tools used in the approach

2.1. Web ontologies

Web ontologies are used to model and share knowledge among various applications in a specific domain facilitating interoperability between different systems. The Web Ontology Language OWL 2 [5] accepted as a World Wide Web Consortium (W3C) is a powerful knowledge representation language; it has been applied successfully for knowledge modelling in many application areas. As a descriptive language, OWL 2 is used to express expert knowledge in a formal way, and as a logical language, it is used to draw conclusions from this knowledge. Every OWL 2 ontology is a machine - processable formal description of a domain of interest and consists of the following three different syntactic categories: *Entities*, *Expressions* and *Axioms*. *Entities*, such as classes, properties, and individuals are identified by IRIs. They form the primitive terms of an ontology and constitute the basic elements of the ontology. *Expressions* represent complex concepts in the domain being described. *Axioms* are statements that are asserted to be true in the domain being described and allow relationships to be established between *Expressions*. The ability to infer additional knowledge is of great importance for designing and deploying OWL ontologies. Sound and complete OWL 2 reasoning is of high complexity, but the OWL 2 new profiles (OWL 2 EL, OWL 2 QL, OWL 2 RL), known also as lightweight ontology languages, restrict the used syntactic categories to improve complexity and practical performance [12]. The profile OWL 2 RL enables the implementation of polynomial time reasoning algorithms using rule-extended database technologies operating directly on RDF triples [12]. As any OWL 2 Ontology can be mapped to RDF, for ontology-based data access can be used SPARQL - a query language across diverse data sources, whether the data is stored natively as RDF or viewed as RDF for RDF models [13].

2.2. A standard for information integration

ISO/IEC 62264 is a multi-part standard that defines the interface content between enterprise activities and control activities [1]. This standard is an agreement between leading companies to create a common framework, and guidelines for design and integration of systems providing common models and terminology for structuring and describing the exchanged information. The standard provides UML models, which are the basis for the development of information systems and standard compliant interfaces between different systems.

The semantic approach, presented in this paper, uses the models and terminology, provided by the standard ISO/IEC 62264 together with a meta-ontology [14], conceptualizing knowledge embedded in the standard as a target OWL ontology in order to use together data presented in different data models. The mapping rules between UML class diagrams (provided by the standard) and OWL elements of the meta-ontology are presented in [15]. The standard conformed

meta-ontology (Fig.1) is comprised of 103 classes, structured in four categories of resources: *Personnel*, *Equipment*, *Material* and *ProcessSegment* (marked on the figure) and five main interface classes, defining the relations Production-Process-Product: *ProductDefinition*, *ProductionCapability*, *ProductionSchedule*, *ProcessSegmentCapability* and *ProductionPerformance*. Based on the main resources, *ProductDefinition* information indicates what must be defined to make a product, *ProductionCapability* information defines what resources are available, *ProductionSchedule* information defines what actual production will be executed and *ProductionPerformance* information states what actual production was achieved.

The meta-ontology has the following favourable features: (i) it establishes clearly defined standardized terminology and structure for general enterprise systems; (ii) it is independent of specific industry or technology; (iii) it facilitates building domain models. The meta-ontology might be used for design of new enterprise systems or reengineering the legacy systems ensuring the interoperability or integration of these systems.

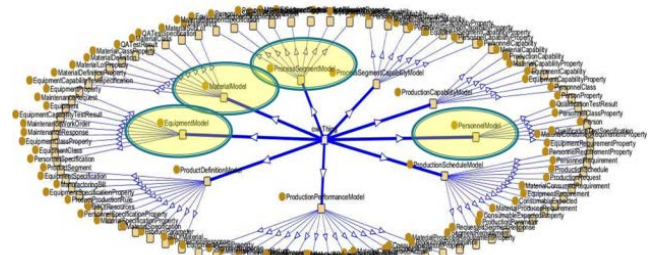


Figure.1: Meta-ontology, based on ISO/IEC 62264

2.3. Software tools, used in the approach

MDD guarantee that business functionality is complete and correct, end-user needs are met and program design supports requirements for scalability, robustness, security, extendibility, and other characteristics, before implementation in code. The process of gathering and analyzing an application's requirements, and incorporating them into a program design, is a complex one and the industry currently supports many methodologies that define formal procedures specifying how to go about it. One characteristic of UML - in fact, the one that enables the widespread industry support that the language enjoys - is that it is methodology-independent. And, using XMI (XML Metadata Interchange), transfer the UML model from one tool into a repository, or into another tool for refinement or the next step in the development process. The so called Object-Relational Mapping (ORM) serves as a bridge between object models, data models and relational databases to automate the transformation between object models and relational data models. The IDE Visual Paradigm 13.0 (<https://www.visual-paradigm.com/>) features all the UML diagrams and Entity Relational Diagrams (ERD) tools essentially in system and database design. Visual Paradigm provides a model driven platform for application development; allows not only to start from creating the models by using UML Class Diagram or Entity Relationship Diagram (ERD) to generating the executable persistence layer from the models, but also to modify the ER model which comes from reverse engineering of an existing database, transform into object model and generate persistence layer. The IDE supports a wide range of databases, including Oracle, DB2, Microsoft SQL Server, PostgreSQL, MySQL and more.

TopBraid Composer Maestro Edition (TBC-ME) (<http://www.topquadrant.com/>) combines world's leading semantic web modeling capabilities with the most comprehensive data conversion options and is a powerful IDE for building, managing and testing configurations of ontologies, for operations on ontologies (merging, alignment, integration, inference, etc.), for automated conversion of spreadsheets, Excel, UML and other data sources, for dynamic connections to relational databases, or running SPARQL queries over relational data.

The D2RQ Platform (<http://d2rq.org/>), integrated in TBC-ME is a matured system for accessing relational databases as virtual, read-only RDF graphs. It offers RDF-based access to the content of relational databases without having to replicate it into an RDF store. The system includes a SPARQL-to-SQL rewriter that can evaluate SPARQL queries over the mapped database.

3. Description of the suggested approach

The approach, suggested in this paper aims to transform, represent and integrate different data and data models into the technological space of ontology engineering for achieving

interoperability between OWL ontologies, relational databases and UML models (Fig.2). At the core of the approach is the usage of reference models, based on the standard for enterprise integration ISO/IEC 62264. One of the benefits is using in a maximum degree the legacy data and data models (relational data models, UML models, Excel data sheets, documents, etc.) presenting them as RDF/OWL structures. Thus the basic advantages of the ontology based system development might be used: intelligent integration, semantic in data management, reasoning upon data and data models, reusable reference models, etc.

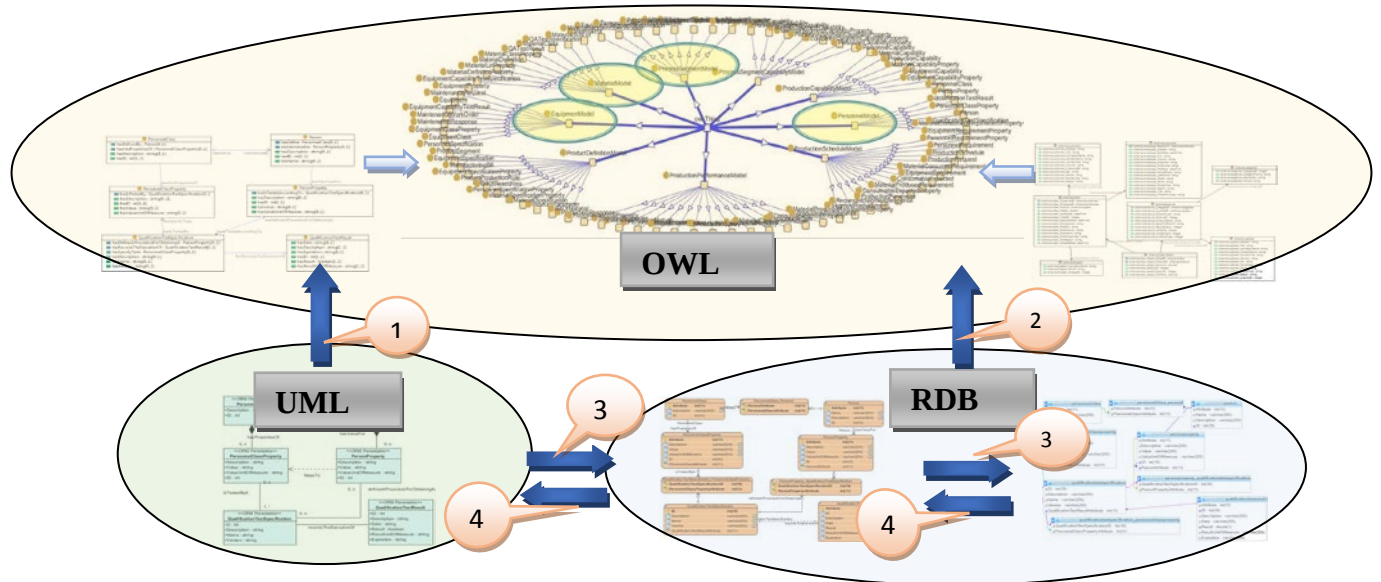


Figure.2: Illustration of the suggested approach

The presented approach is realized with two mutual commercial software IDEs for automatic and personalized model transformations: Visual Paradigm 13.0 for UML and ERD transformations and Top Braid Composer ME 4.2.0 used for ontology engineering. As a DBMS MySQL is used and the data connection is done with the free software tool to handle the administration of MySQL over the Web: phpMyAdmin (<https://www.phpmyadmin.net/>). The approach states on the usage of a reference standard based meta-ontology as a target ontology based on the models and terminology, provided by the standard ISO/IEC 62264.

4. Applicability of the suggested approach

The realized model transformations are pointed on the figures with the numbers 1-4.

4.1. UML class diagram to OWL ontology

The transformation from UML class diagram (Visual Paradigm) to OWL ontology (TopBraid Composer) is pointed with (1) on Figure 2. One of the main resource models of ISO/IEC 62264 is used as it is presented in the standard – the Personnel model. The Personnel model contains the information about specific personnel, classes of personnel, and qualifications of personnel. The *PersonnelClass* is a class to describe a grouping of persons with similar characteristics. Each *PersonnelClass* may have zero or more recognized properties. The class *Person* represents a specifically identified individual. A person may be a member of zero or more personnel classes. These specify the current property values of the person for the associated personnel property. A *qualification test specification* may be associated with a *personnel class property* or *person property*. A *qualification test result* records the results from a qualification test for a specific person. Using a model-based transformation approach, implemented in TopBraid Composer ME 4.2.0, based on SPARQL Rules, XMI model of the UML Personnel model, created in Visual Paradigm 13.0 is converted to OWL

(Figure 3). The transformation is not still personalized, the names of the classes and properties are generated automatically.

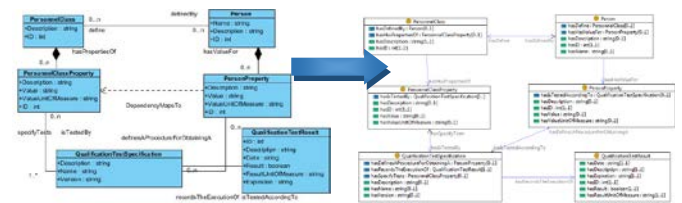


Figure.3: Transformation from UML class diagram to OWL ontology

4.2. Relational database to OWL ontology

The transformation of a relational database (PhpMyAdmin) to OWL ontology (TopBraid Composer) is pointed with (2) on Figure 2. The transformation is done (Figure 4) with the integrated D2RQ mapping language in the TopBraid Composer ME 4.2.0.



Figure.4: Transformation from relational database to OWL ontology

A sample MySQL database for Inventory/Orders/Customers system - northwind.sql is used and with D2RQ mapping language a static import of schema and dynamic import of data is done. The schema and the data from the used database might be used along with a domain ontology which can be integrated into the meta-

ontology for *PersonnelModel* or *ProductionSchedulingModel*, or a suitable mapping directly to the meta-ontology might be done. SPARQL queries to the content of the relational database can be performed without having to replicate it in ontology data store.

4.3. Object-Relational Mapping

The transformation of an UML class diagram (Visual Paradigm) to ERD data model (Visual Paradigm) and to MySQL (PhpMyAdmin) is pointed with (3) on Figure 2. One of the main resource models of ISO/IEC 62264 is used as it is presented in the standard – the Personnel model. The UML Personnel model, created in Visual Paradigm 13.0 is converted to ERD physical model and a database Personnel.sql is generated. The UML class diagram, represented by

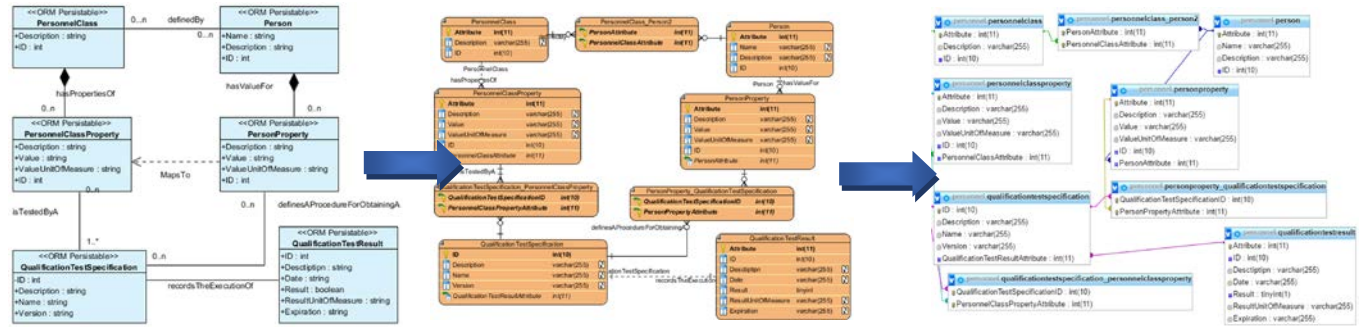


Figure 5.: Transformation from UML class diagram to ERD data model and to MySQL database

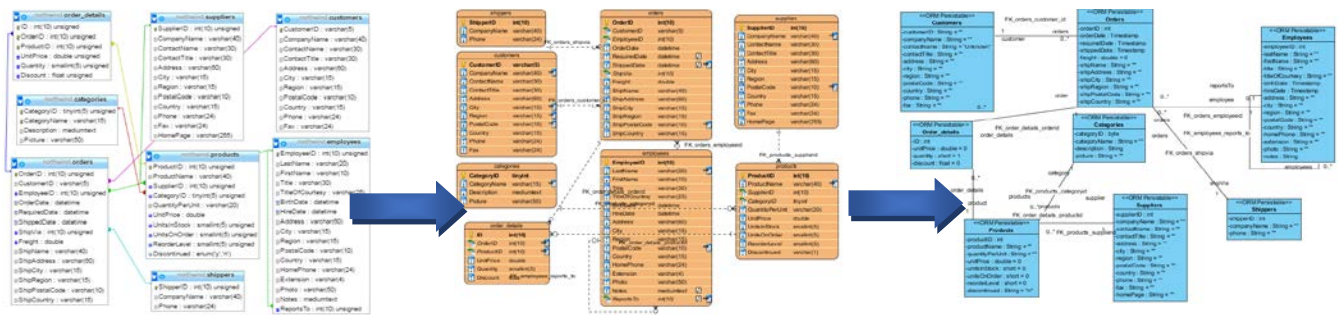


Figure 6.: Transformation from relational database to ERD data model and to UML class diagram

5. Conclusions

The suggested approach, based on the joint use of standards for integration, relational databases, UML models and OWL ontologies aims to reduce the effort, cost, and errors during the design, implementation and integration of systems. Ontologies ensure easy merging, extending and sharing of heterogeneous data models, rich semantics, and reusable modules. Semantics shows enormous potential in making software systems more efficient, adaptive and intelligent. Ontology-based systems allow humans and computer systems to exchange knowledge without ambiguity and also makes it possible to use logical deduction to infer additional information from the facts stated explicitly in ontologies.

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A MAPREDUCE SOLUTION FOR HANDLING LARGE DATA EFFICIENTLY

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Abstract: Big data is large volume, heterogeneous, distributed data. Big data applications where data collection has grown continuously, it is expensive to manage, capture or extract and process data using existing software tools. With increasing size of data in data warehouse it is expensive to perform data analysis. In recent years, numbers of computation and data intensive scientific data analyses are established. To perform the large scale data mining analyses so as to meet the scalability and performance requirements of big data, several efficient parallel and concurrent algorithms got applied. For data processing, Big data processing framework rely on cluster computers and parallel execution framework provided by MapReduce. MapReduce is a parallel programming model and an associated implementation for processing and generating large data sets. In this paper, we are going to work around MapReduce, use a MapReduce solution for handling large data efficiently, its advantages, disadvantages and how it can be used in integration with other technology.

Keywords: DATA MINING; BIG DATA; CLUSTERING; PARALLEL PROCESSING; HADOOP; HDFS; MAPREDUCE;

1. Introduction

Data Mining is analyzing the data from different perspectives and summarizing it into useful information that can be used for business solutions and predicting the future trends. Mining the information helps organizations to make knowledge driven decisions. Data mining (DM), also called Knowledge Discovery in Databases (KDD), is the process of searching large volumes of data automatically for patterns such as association rules. It applies many computational techniques from statistics, information retrieval, machine learning and pattern recognition. Data mining extract only required patterns from the database in a short time span. Based on the type of patterns to be mined, data mining tasks can be classified into summarization, classification, clustering, association and trends analysis [1]. Among several techniques in data mining, clustering is the main considerable point which is used to retrieve the essential knowledge from the very huge collection of data. Clustering can handle both the complicated and very large datasets which can be of diverse data types. To classify the large data sets clustering is the vital solution [2]. Big Data is a new term used to identify the data sets that are of large size and have greater complexity [3]. Big data is defined as large amount of data which requires new technologies and architectures to make possible to extract value from it by capturing and analysis process [4].

Over the last few years, MapReduce has emerged as the most popular computing paradigm for parallel, batch style and analysis of large amounts of data. Many areas where massive data analysis is required, MapReduce are used. There are evolving numbers of applications that handle big data, but to handle such huge collection of data is a very challenging problem today. Here, we got the MapReduce or its open source equivalent Hadoop which is a powerful tool for building such applications [5]. MapReduce divides the computational flow into two main phases: **Map** and **Reduce**. By simply designing Map and Reduce functions, developers are able to implement parallel algorithms that can be executed across the cluster [6]. The steps involved in working of MapReduce can be shown in as **figure 1** [7]:

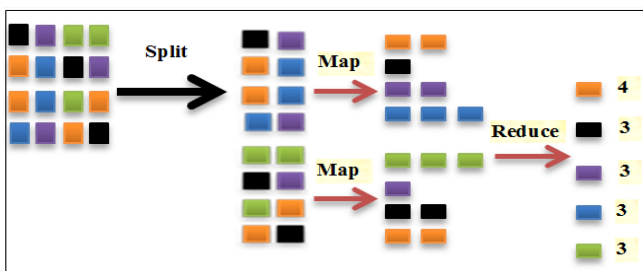


Fig.1. Steps in MapReduce to process the database

Data intensive processing is fast and currently becoming a necessity to handle the large databases efficiently. It is required to design algorithms that must be capable of scaling to real world datasets. MapReduce is a programming model which is inspired by functional programming which allows expressing distributed computations on massive amounts of data. It is designed for large scale data processing as it allows running on clusters of commodity hardware [8]. MapReduce is a powerful parallel programming technique for distributed processing of vast amount of data on clusters [9].

2. Background

In this section, we review the Apache Hadoop Fundamentals (HDFS and MapReduce) Explained with a Diagram:

2.1. Hadoop Framework

Apache Hadoop is an open source Java framework for processing and querying vast amounts of data on large clusters of commodity hardware. With a significant technology investment by Yahoo!, Apache Hadoop has become an enterprise ready cloud computing technology. It is becoming the industry defacto framework for Big Data processing. Hadoop impact can be boiled down to four salient characteristics. Hadoop enables scalable, costeffective, flexible, fault tolerant solutions [10]. Hadoop was derived from Google's MapReduce and Google File System (GFS) [11]. Hadoop has **two** primary components, namely, **HDFS** and **MapReduce** programming framework. The most significant feature of Hadoop is that HDFS and MapReduce are closely related to each other. Therefore, the storage system is not physically separated from the processing system [12]. The following **figure 2** shows the Hadoop framework main components [13].

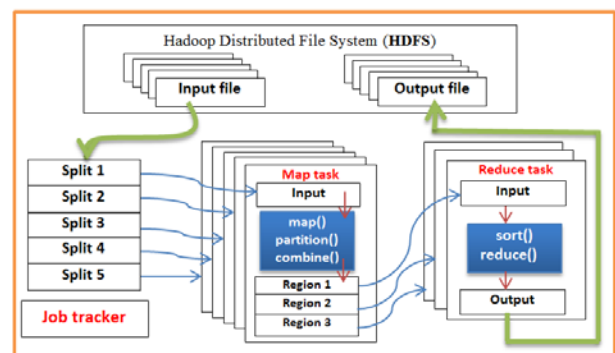


Fig.2. Hadoop Framework

2.1.1. HDFS (Hadoop Distributed File System)

HDFS stands for Hadoop Distributed File System, it is the

storage system used by Hadoop. The following is a high level architecture that explains how HDFS works(see figure 3) [21].

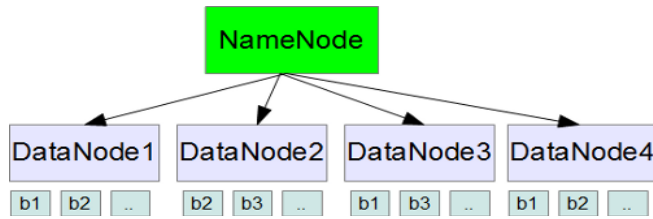


Fig.3. HDFS Diagram.

- In the above diagram, there is one NameNode, and multiple DataNodes(servers). b1, b2, indicates data blocks.
- When you dump a file (or data) into the HDFS, it stores them in blocks on the various nodes in the hadoop cluster. HDFS creates several replications of the data blocks and distributes them accordingly in the cluster in way that will be reliable and can be retrieved faster. A typical HDFS block size is 128MB. Each and every data block is replicated to multiple nodes across the cluster.
- Hadoop will internally make sure that any node failure will never results in a data loss.
- There will be one NameNode that manages the file system metadata.
- There will be multiple DataNodes (These are the real cheap commodity servers) that will store the data blocks.
- When you execute a query from a client, it will reach out to the NameNode to get the file metadata information, and then it will reach out to the DataNodes to get the real data blocks
- Hadoop provides a command line interface for administrators to work on HDFS.
- The NameNode comes with an in-built web server from where you can browse the HDFS filesystem and view some basic cluster statistics.

2.1.2. MapReduce Framework

MapReduce is a software framework introduced by Google in 2004 to support distributed computing on large data sets on clusters of computers. MapReduce is a programming model for processing and generating large data sets. Users specify a map function that processes a key/value pair to generate a set of intermediate key/value pairs and a reduce function that merges all intermediate values associated with the same intermediate key [11]. MapReduce is designed to be used by programmers, rather than business users. It is a programming model, not a programming language. It has gained popularity for its easiness, efficiency and ability to control Big Data in a timely manner [7]. Google proposed the MapReduce programming framework based on functional programming. The framework divides the work into independent tasks and parallelizes the computation flow across large scale clusters of machines, taking care of communications among them and possible failures, and efficiently handling network bandwidth and disk usage [6]. The following is a high-level architecture that explains how MapReduce works (see figure 4) [21].

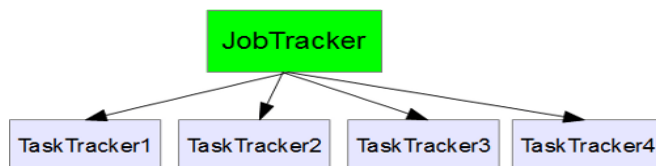


Fig.4. MapReduce Diagram

- MapReduce is a parallel programming model that is used to retrieve the data from the Hadoop cluster
- In this model, the library handles lot of messy details that programmers doesn't need to worry about. For example, the library takes care of parallelization, fault tolerance, data distribution, load balancing, etc.
- This splits the tasks and executes on the various nodes parallelly, thus speeding up the computation and retriving required data from a huge dataset in a fast manner.

- This provides a clear abstraction for programmers. They have to just implement two functions: map and reduce.
- The data are fed into the map function as key value pairs to produce intermediate key/value pairs
- Once the mapping is done, all the intermediate results from various nodes are reduced to create the final output
- JobTracker keeps track of all the MapReduces jobs that are running on various nodes. This schedules the jobs, keeps track of all the map and reduce jobs running across the nodes. If any one of those jobs fails, it reallocates the job to another node, etc. In simple terms, JobTracker is responsible for making sure that the query on a huge dataset runs successfully and the data is returned to the client in a reliable manner.
- TaskTracker performs the map and reduce tasks that are assigned by the JobTracker. TaskTracker also constantly sends a heartbeat message to JobTracker, which helps JobTracker to decide whether to delegate a new task to this particular node or not.

3. Mapreduce Architecture

In most computation related to high data volumes, it is observed that two main phases are commonly used in most data processing components this is shown in below figure 5. Map Reduce created an abstraction phases of Map Reduce model called **mappers** and **reducers**. When it comes to processing large data sets, for each logical record in the input data it is often required to use a mapping function to create intermediate key value pairs. Then another phase called reduce to be applied to the data that shares the same key, to derive the combined data appropriately [14].

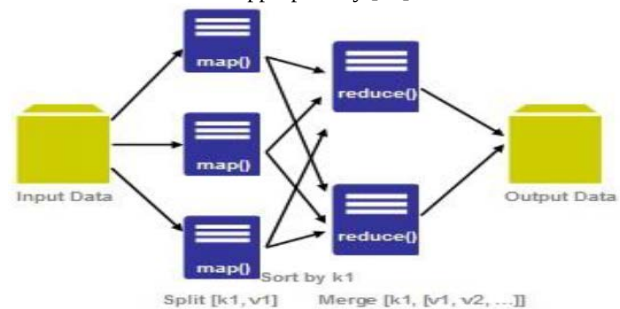


Fig.5. MapReduce Architecture

Mapper

The mapper is applied to every input key-value pair to generate an arbitrary number of intermediate key-value pairs. The standard representation of this is as follows:

$\text{map}(\text{inKey}, \text{inValue}) \rightarrow \text{list}(\text{intermediateKey}, \text{intermediateValue}).$

The purpose of the map phase is to organize the data in preparation for the processing done in the reduce phase. The input to the map function is in the form of key-value pairs, even though the input to a MapReduce program is a file or file(s). By default, the value is a data record and the key is generally the offset of the data record from the beginning of the data file. The output consists of a collection of key-value pairs which are input for the reduce function. The content of the key-value pairs depends on the specific implementation.

For example, a common initial program implemented in MapReduce is to count words in a file. The input to the mapper is each line of the file, while the output from each mapper is a set of key-value pairs where one word is the key and the number 1 is the value.

$\text{map}: (\text{k1}, \text{v1}) \rightarrow [(\text{k2}, \text{v2})]$

The file_name and the file_content which is denoted by k1 and v1. So, with in the map function user may emit the any arbitrary key/value pair as denoted in the list [k2, v2]. To optimize the processing capacity of the map phase, MapReduce can run several identical mappers in parallel. Since every mapper is the same, they produce the same result as running one map function.

Reducer

The reducer is applied to all values associated with the same intermediate key to generate output key-value pairs.

```

reduce(intermediateKey,list(intermediateValue))-->
list(outKey, outValue)

```

Each reduce function processes the intermediate values for a particular key generated by the map function and generates the output. Essentially there exists a one-one mapping between keys and reducers. Several reducers can run in parallel, since they are independent of one another. The number of reducers is decided by the user. By default, the number of reducers is 1. Since we have an intermediate group by operation, the input to the reducer function is a key value pair where the key- k2 is the one which is emitted from mapper and a list of values [v2] with shares the same key.

reduce: (k2 , [v2]) → [(k3 , v3)] .

4. Programming Model

MapReduce is a programming model designed for processing large volumes of data in parallel by dividing the work into a set of independent tasks. The nature of this programming model and how it can be used to write programs which run in the Hadoop environment is explained by this model. Hadoop is an open source implementation of this environment [15].

The computation takes a set of input key/value pairs, and produces a set of output key/value pairs. The user of the MapReduce library expresses the computation as two functions: map and reduce. Map, written by the user, takes an input pair and produces a set of intermediate key/value pairs. The MapReduce library groups together all intermediate values associated with the same intermediate key I and passes them to the reduce function. The reduce function, also written by the user, accepts an intermediate key I and a set of values for that key. It merges these values together to form a possibly smaller set of values. Typically just zero or one output value is produced per reduce invocation. The intermediate values are supplied to the user's reduce function via an iterator. This allows us to handle lists of values that are too large to fit in memory. MapReduce [22] requires that the operations performed at the reduce task to be both associative and commutative. This two stage processing structure is illustrated in

Figure 6:

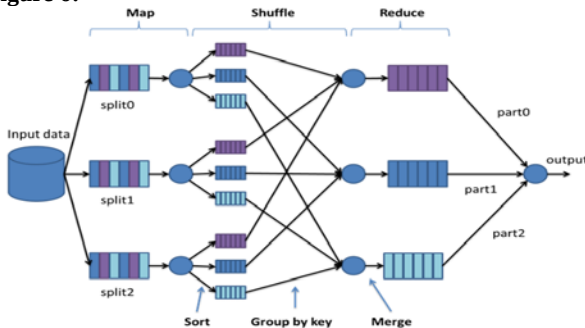


Fig.6. MapReduce simplified flowchart

Map and Reduce are two functions. The main job of these two functions are sorting and filtering input data. During Map phase data is distributed to mapper machines and by parallel processing the subset it produces <key, value>pairs for each record. Next shuffle phase is used to repartition and sorting that pair within each partition. So the value corresponding same key grouped into {v1, v2,...} values. Last during Reduce phase reducer machine process subset <key, {v1, v2,>pairs parallel in the final result is written to distributed file system [15]. A Hadoop MapReduce program also has a component called the Driver. The driver is responsible for initializing the job with its configuration details, specifying the mapper and the reducer classes for the job, informing the Hadoop platform to execute the code on the specified input file(s) and controlling the location where the output files are placed [14].

4.1 Example

Consider the problem of counting the number of occurrences of each word in a large collection of documents. The user would write code similar to the following pseudocode.

```

map(String key, String value):

```

```

// key: document name
// value: document contents
for each word w in value:
    EmitIntermediate(w, "1");
reduce(String key, Iterator values):
    // key: a word
    // values: a list of counts
    int result = 0;
    for each v in values:
        result += ParseInt(v);
    Emit(AsString(result));

```

The map function emits each word plus an associated count of occurrences (just '1' in this simple example). The reduce function sums together all counts emitted for a particular word. In addition, the user writes code to fill in a mapreduce specification object with the names of the input and output files, and optional tuning parameters. The user then invokes the MapReduce function, passing it the specification object. The user's code is linked together with the MapReduce library (implemented in Java) [16].

4.2 Types

Even though the previous pseudocode is written in terms of string inputs and outputs, conceptually the map and reduce functions supplied by the user have associated types.

```

map (k1,v1) →list(k2,v2)
reduce (k2,list(v2)) →list(k3,v3).

```

That is, the input keys and values are drawn from a different domain than the output keys and values. Furthermore, the intermediate keys and values are from the same domain as the output keys and values. Finally we can say the Input and Output types of a MapReduce job as:

```

(input) <k1, v1> -> map -> <k2, v2> -> combine -> <k2, v2> -
-> reduce -> <k3, v3> (output).

```

5. Problem Formulation

MapReduce over traditional DBMS: Our traditional DBMSs have adopted such strategies which are not appropriate for solving extremely large scale data processing tasks. There has been a need for some special purpose data processing tools that are adapted for solving such problems[17]. While MapReduce is referred to as a new way of processing big data in data center computing, it is also criticized as a "major step backwards" in parallel data processing in comparison with DBMS [18]. However, many MapReduce proponents in industry argue that MapReduce is not a DBMS and such between them is not a just [19]. Although Hadoop won the 1st position in GraySort benchmark test for 100 TB sorting (1 trillion 100-byte records) in 2009, its winning was achieved with over 3,800 nodes. MapReduce or Hadoop would not be a cheap solution if the cost for constructing and maintaining a cluster of that size was considered [20]. Analysis of 10-months of MR logs from Yahoo's M45 Hadoop cluster and MapReduce usage statistics at Google are also available. The studies exhibit a clear tradeoff between efficiency and fault-tolerance. MapReduce increases the fault tolerance of long-time analysis by frequent checkpoints of completed tasks and data replication. However, the frequent I/Os required for fault tolerance reduce efficiency. Parallel DBMS aims at efficiency rather than fault tolerance. DBMS actively exploits pipelining intermediate results between query operators. However, it causes a potential danger that a large amount of operations need be redone when a failure happens. With this fundamental difference, we describe some advantages and limitations of MapReduce framework [5].

6. Advantages of Mapreduce

MapReduce is simple and efficient for computing aggregate. Thus, it is often compared with "filtering then group-by aggregation" query processing in a DBMS. Here are major advantages of the MapReduce framework for data processing. For the huge data processing task, the key advantages of the MapReduce framework are as [5]:

- Simple and easy to use:** The MapReduce model is simple but expressive. A programmer defines his task by using only Map and Reduce functions. There is no need for him to specify the physical distribution of his work across nodes.
- Flexible MapReduce:** It does not have any dependency on data model and schema. A programmer can deal with irregular or unstructured data more easily than they do with DBMS.
- Independent of the storage:** It is independent from underlying storage layers. Thus, MapReduce can work with different storage layers such as Big Table and others.
- Fault tolerance:** It is highly fault-tolerant. It is reported that MapReduce can continue to work in spite of an average of 1.2 failures per analysis job at Google.
- High scalability:** The best advantage of using MapReduce is high scalability. Yahoo! reported that their Hadoop gear could scale out more than 4,000 nodes in 2008.

MapReduce is simple and efficient tool for query processing in a DBMS. Thus, due to these major advantages of MapReduce, we have integrated it with one of the advance technique of data processing from large databases. The increasing interest and popularity of MapReduce has led some relational DBMS vendors to support MapReduce functions inside the DBMS. This capability not only offers the benefits outlined above for deploying user defined functions, but also adds the advantages of MapReduce to the relational DBMS environment, i.e., the ability to process multi-structured data using SQL. It also brings the maturity of relational DBMS technology to MapReduce processing. The Teradata Aster Database is an example of a product that supports MapReduce [18].

7. Discussion And Challenges

MapReduce is becoming ubiquitous, even though its efficiency and performance are controversial. There is nothing new about principles used in MapReduce. However, MapReduce shows that many problems can be solved in the model at scale unprecedented before. Due to frequent checkpoints and runtime scheduling with speculative execution, MapReduce reveals low efficiency. However, such methods would be necessary to achieve high scalability and fault tolerance in massive data processing. Thus, how to increase efficiency guaranteeing the same level of scalability and fault tolerance is a major challenge. The efficiency problem is expected to be overcome in two ways: improving MapReduce itself and leveraging new hardware. How to utilize the features of modern hardware has not been answered in many areas. However, modern computing devices such as chip-level multiprocessors and Solid State Disk can help reduce computations and I/Os in MapReduce significantly. The size of MR clusters is continuously increasing. A 4,000-node cluster is not surprising any more. How to efficiently manage resources in the clusters of that size in multi-user environment and achieving high utilizations of MR clusters is also challenging [5].

8. Conclusion

We are living in the big data era where enormous amounts of heterogeneous, semi structured and unstructured data are continually generated at unprecedented scale, and processing large volumes of data has never been greater. Through better Big Data analysis tools like Map Reduce over Hadoop and HDFS, guarantees faster advances in many scientific disciplines and improving the profitability and success of many enterprises. MapReduce has received a lot of attentions in many fields, including data mining, information retrieval, image retrieval, machine learning, and pattern recognition. However, as the amount of data that need to be processed grows, many data processing methods have become not suitable or limited.

In this paper we discussed about the MapReduce framework for efficient analysis of big data and for solving challenging data processing problems on large scale datasets in different domains. MapReduce provides a simple way to scale your application. It

effortlessly scales from a single machine to thousands, providing fault tolerant & high performance.

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ADVANCED CREATING OF 3D DENTAL MODELS IN BLENDER SOFTWARE

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Abstract: The development of virtual 3D dental models evolves with great pace. This is a stage of construction, which is of great importance in future developments of real manipulation, prosthetics and other activities set for execution. Concept of modeling of 3D dental models is determined on the basis of concrete requirements, factors and opportunities. A good basis for creating virtual dental models provide Blender software combined with specialized applications which improve the process of modeling.

Keywords: Blender, 3D, dental, tooth, implants, open source

1. Problem discussion

The modeling of 3D virtual models of dental models is particularly relevant. This is done in regardless of, and by means of computer-generated environment to examine the dental specimens for purposes of science. The developed 3D models are used by designers, engineers, medical specialists, etc. Designing 3D virtual dental models is carried out through the use of methodologies for the relevant software systems. The following types of design are possible: by individual design using design tools and/or by generating parametric virtual models.

The aim of this study is to determine the correct approach to develop 3D virtual dental models which have the necessary qualities, using the resources of modern open source program Blender [1, 2].

2. Objective and research methodologies

A standard method for developing 3D virtual dental models of teeth, jaw tissues, dental implants, abutments and dental crowns in Blender software is to use set of tools for modeling, editing, modification and sculpturing (fig. 1) [3 - 13].

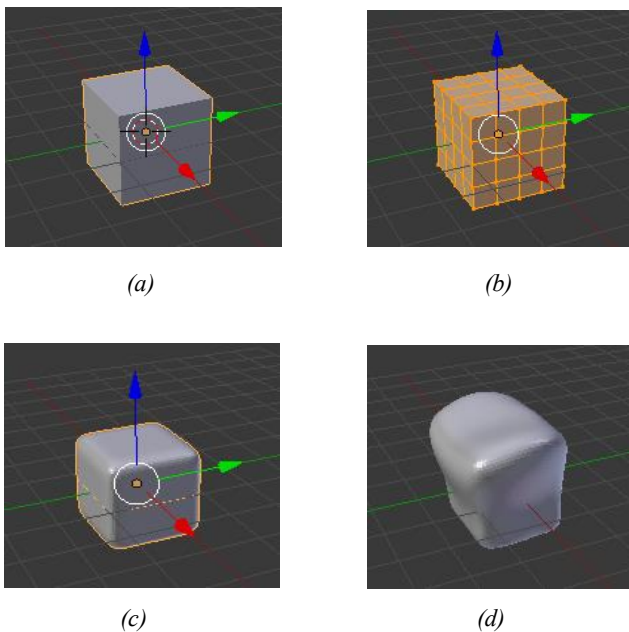


Fig.1. A standard method for developing 3D virtual dental via toolbox of Blender software (a) object mode; (b) edit mode; (c) modifier; (d) sculpt mode

If necessary the set of tools in Blender software can be used combined with parametric built virtual dental models. The

parametric building of virtual dental models is through specialized applications addons. These are applications that are specifically designed for the needs of 3D modeling of teeth, jaws, tissue and dental implants, abutments and dental crowns. At this stage the applications running on the latest version 2.76 Blender's are: Human teeth addon и Open Dental CAD addon [14, 15].

Human teeth addon enables direct modeling of high-quality 3D models of teeth. The virtual models are accurately modeled and tailored to the geometric characteristics of such real samples. Computer-generated full set of teeth is shown on fig. 2. The green box has covered the location that occupies Human teeth addon in the composition of Blender software.

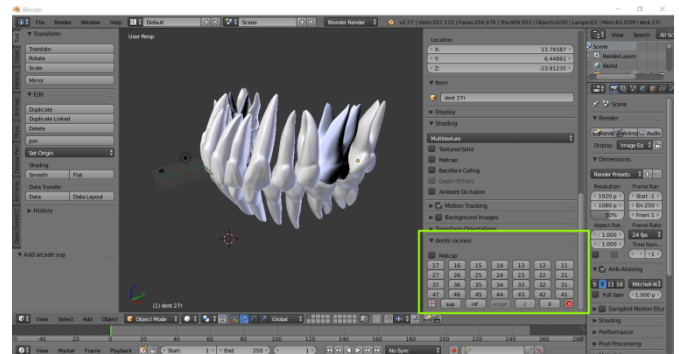


Fig. 2. Interface of Blender software with enabled application Human teeth addon. There is a computer-generated full set of teeth on the desktop

The possibilities for parametric modeling are: individual computer generating of tooth (by number) or multiple computer modeling using "sup" for upper dentition and "in" for lower dentition. The individual numbering of teeth is shown on fig. 3.

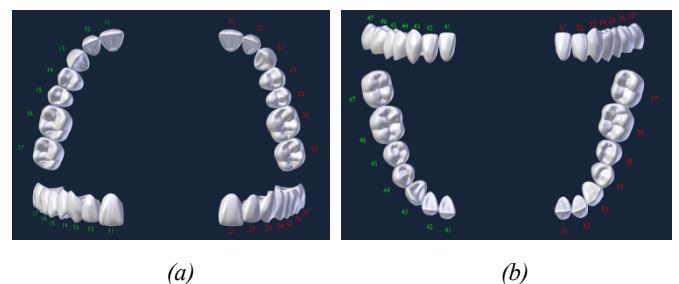


Fig. 3. Individual numbering of teeth (a) upper dentition (b) a lower dentition

The teeth are modeled with high quality they have complied with all features. Fig. 4 shows part of the models of teeth in schematic options and 3D prepared [14].

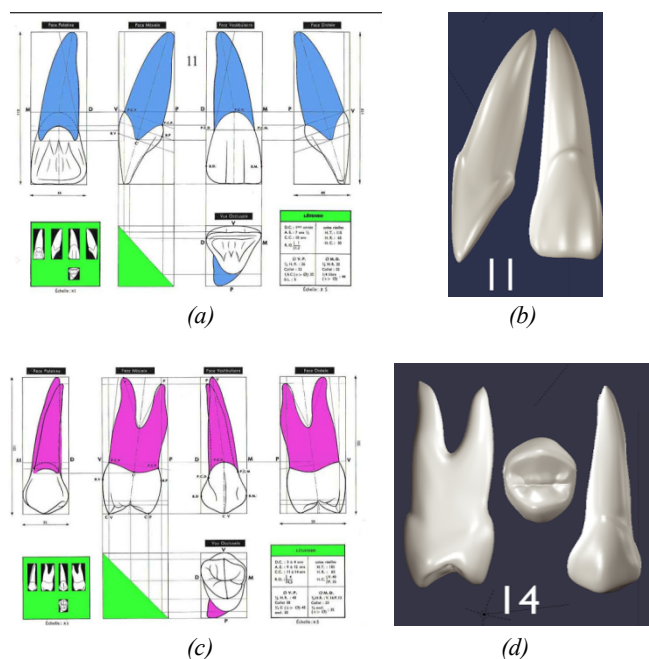


Fig.4. Dental models (a) scheme of tooth number 11; (b) 3D model of tooth 11; (c) diagram of tooth number 14 (d) model tooth 14

Once the computer-generated model of the respective tooth is done the designer can make individual remodeling (fig.5).

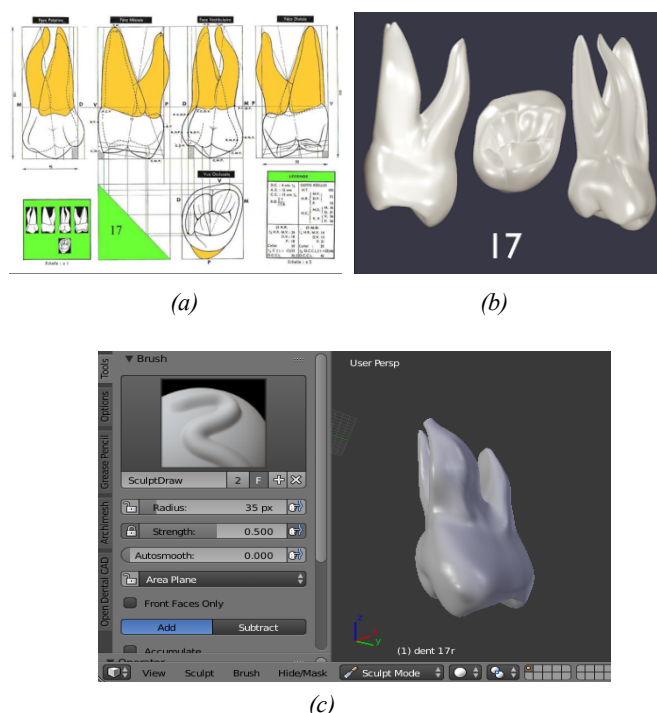


Fig.5. Model of tooth number 17 (a) scheme of tooth No.17; (b) 3D model of tooth 17 by default; (c) 3D model of tooth 17 sculpt modified

Human teeth addon application can be provided by download from the website <http://byaapplication3d.blogspot.bg/p/ents-3d.html>. When used the Human teeth addon in combination with Open Dental CAD addon the opportunities to build 3D computer models dental are significantly increasing [15]. Fig. 6 shows the

operation panel of the Open Dental CAD addon in Blender's environment.

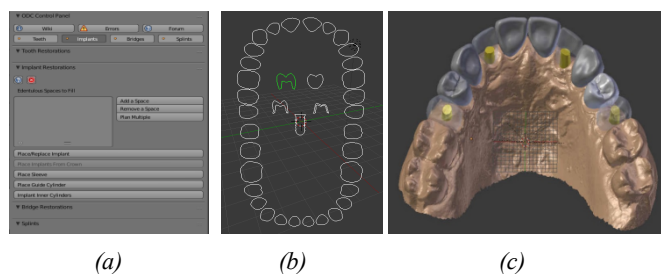


Fig.6. Model of tooth number 17 (a) scheme of tooth No.17; (b) 3D model of tooth 17 by default; (c) 3D model of tooth 17 sculpt modified

The application Open Dental CAD addon can be provided by download from the webpage https://github.com/patmo141/odc_public/wiki.

3. Conclusion

Very well constructed variations of dental and implant 3D computer models not only from a visual standpoint, but also as a foundation on which to build, modify and alter depending on the job that needs to be realized is an opportunity that is successfully implemented in development of projects related to high-quality dental visualizations. Fully-functional applications Human teeth addon and Open Dental CAD addon are freely available for providing and integration into practice. Dental applications integrated into Blender software provide very good perspective in the workflow.

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PHASE TRANSITION – MATHEMATICS AND MATHEMATICAL PHYSICS IN MICRO-FOUNDRY

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Abstract: This article presents fundamental results in mathematics and mathematical physics on the example of a theoretical model of structure formation in casting. Basic scientific results are innovations for all micro-foundries.

Keywords: FUNDAMENTAL RESULTS, MATHEMATICS & MATEMATCAL PHYSICS, INOVATIONS, MICRO-FOUNDRY

1. Introduction – Phase transitions

1.1 Equilibrium thermodynamics

Basde states of matter and physical processes of the phase transitions are shown in Fig. 1 Physics • Phase transitions:

a) Fundamental states of mater and different phase transitions; b) Phase diagram a single component system in coordinates P–T; c) thermodynamics driving force of phase transition – crystallization and growth: vapor–crystal, and liquid–crystal; d) quantum phase transition of second order; e) solidification of Aluminum alloy – phase transition of first order

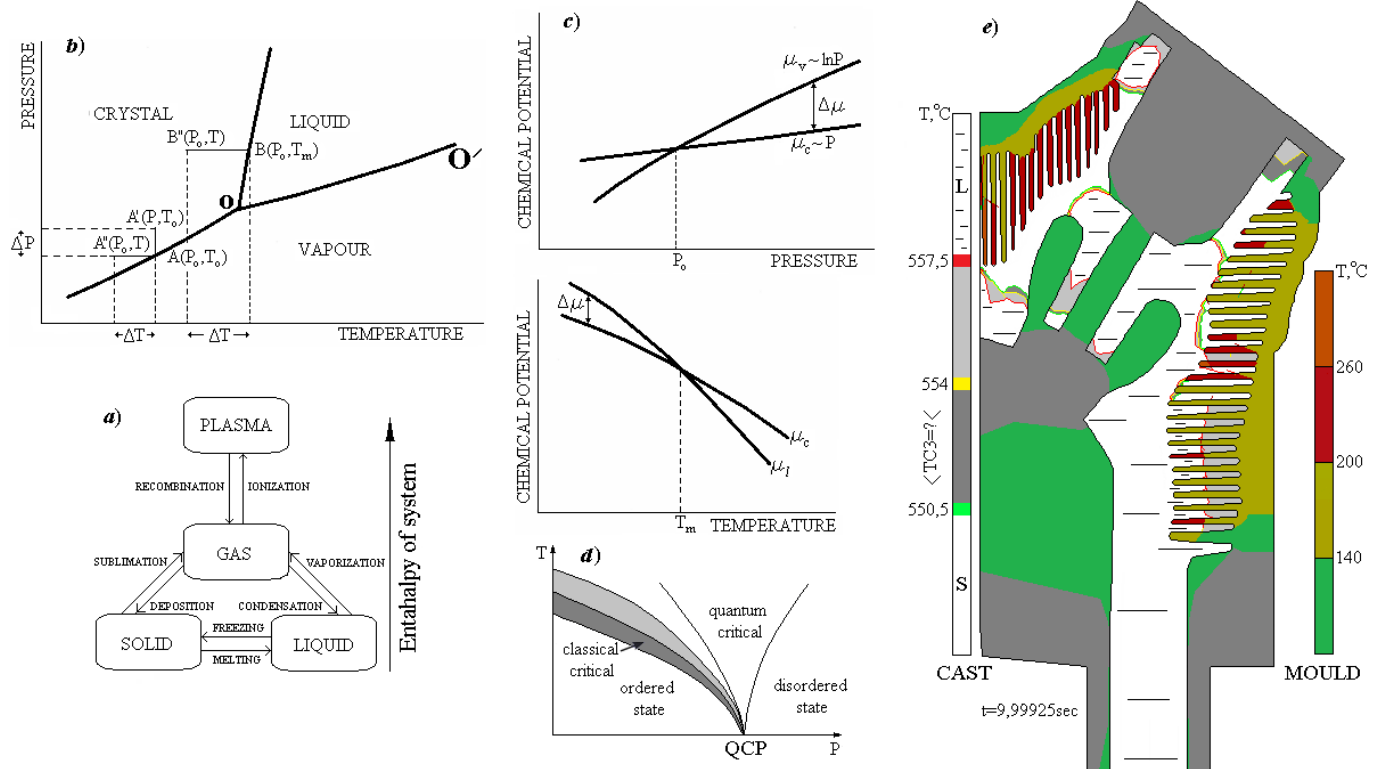


Fig. 1 Phase Transition: a) Fundamental states of the matter and different phase transition processes; b) Phase diagram a single component system [13]; c) thermodynamic diving force and crystallization of liquid [13]; d) Phase diagram of a second order quantum phase transition; Solidification process – zone of phase transition.

Thermodynamics system: **I. First law** – the internal energy (U) = heat quantity (q) + work (w); **II. Second law**: Impossible process, the only end result of which will be conversion in work the heat, take away from the source with identical temperature everywhere. (**Kelvin postulate**); Impossible process, the only end result, of which would be transfer of the heat from body with given temperature to body with higher temperature (**Clausius postulate**). The system of the state with temperature is changing by giving or receiving an infinitesimal amount of heat δQ for the infinitesimal time is quantity $\delta Q/T$. For **cyclic process** of the system with finite number processes $(\delta Q/T)_i$, $i=1, 2, 3, \dots$, we have for irreversible cyclic process $\sum_{i=1}^n (\delta Q/T)_i \leq 0$ and $\sum_{i=1}^n (\delta Q/T)_i = 0$ for reversible cyclic process. At infinite number reversible processes $\Rightarrow$

the sum Σ is replaced with integral on close curve $\oint dQ/T \leq 0$ интеграл по затворена линия i.e. for irreversible $\oint dQ/T \leq 0$

and reversible $\oint dQ/T = 0$ process. The value of the integral

for reversible process das not depend on the path. For reversible process between two equilibrium states A and B we have $\left(\int_A^B dQ/T \right)_{I_{AB}} = \left(\int_A^B dQ/T \right)_{II_{BA}}$. The values of the integrals I_{AB} and II_{BA}

are equivalents, but $(-II_{BA})$ have negative sign. The properties (**equality**) $I_{AB} = II_{BA}$ determined new function of the state of the system termed **entropy** S . For reversible process between an **initial equilibrium standard fixed state** O and finite equilibrium state A we

have $S(A) = \int_0^A dQ/T$. Change of S for a time of infinitesimal reversible process is resulting from division of the quantity of heat absorbed by the system of the system temperature:

$$dS = dQ/T. \quad (\text{Entropy, A})$$

Entropy is determined to additive constant. Single-phase system there is only one homogeneous substance. Heterogeneous system is composed of different homogeneous parts. The Gibbs equation for the rule of the phases is

$$v = 2 + n - f,$$

where v – degree of variability or number the degree of the freedom; n – independents number of components; f – number of the phases. **Thermodynamic potentials** of the system are: Enthalpy H , Helmholtz function F and Gibbs function

$$H = U + pV, F = U - TS \text{ и } G = H - TS, \quad (\text{I, 1, 2, 3})$$

where p , V – pressure and volume of the system. Thermodynamic base equations introduced in differential form are:

$$\begin{aligned} dU &= TdS - PdV, & dH &= TdS + VdP, \\ dF &= -SdT - PdV, & dG &= -SdT + VdP. \end{aligned} \quad (\text{II, 1, 2, 3, 4})$$

Thermodynamic equations (II) for multicomponent system are:

$$\begin{aligned} dU &= TdS - PdV + \sum_j \mu_j dN_j, & dH &= TdS + VdP + \sum_j \mu_j dN_j, \\ dF &= -SdT - PdV + \sum_j \mu_j dN_j, & dG &= -SdT + VdP + \sum_j \mu_j dN_j, \end{aligned} \quad (\text{III, 1, 2, 3, 4}).$$

where $\mu_j = (\partial U / \partial N_j)_{V,S} = (\partial F / \partial N_j)_{V,T} = (\partial G / \partial N_j)_{p,T} = (\partial U / \partial N_j)_{p,S}$ –

chemical potential of j -component; N_j – number of molecules of the component. With partial derivatives in μ_j is obtained Maxwell relation and with eq.(III) can obtain thermodynamic equation of the state. **III. Third law:** *entropy of every system at the absolute zero always can be considered zero. (Nernst theorem).* Thus, it can be assumed that at $T = 0$ K entropy of the default state of the system O and used to determine the additive constant entropy. Thermodynamic state of a large number of dynamic conditions (particles) in **statistical mechanics** probability π . In an isolated system spontaneously occur only those processes that bring the system most likely (most sustainable) state compatible with the full power of the system. Boltzmann establish a functional link between the probability π and entropy S

$$S = k \ln \pi + \text{const} \quad (\text{Entropy, B})$$

where Boltzmann constant is $k = R/A$, R – gaseous constant, A – Avogadro number. For given thermodynamic state π is not probability, and great (∞) number dynamical states create major difficulties which are removed in the classical case by: 1. Dynamic states of multiple systems form (∞^{2v}), where v – the number of the system freedom; 2. Therefore all state can be presented with point in $2v$ -dimensional space – phase space. мерного пространство; 3. The phase space is divided into small cells $\langle\langle\text{over}\rangle\rangle$ -volume (hyper-volume) τ and the points covered in it are described by one state. Dynamic state, represented by cells introduces a break in the concept of state of a system, which allows calculation of π methods of statistical analysis and hence statistical definition of entropy. It is noted that π and S depend of the choice of the size cells τ ; if $\tau \rightarrow 0$ it π and S is turn in ∞ . It shows that in case of τ in π appears in a multiplier and uncertainty π leads to uncertainty and an S i.e. classical statistical mechanics cannot result in constants entropy. Randomness associated with the π , and for this and with S , in the classical interpretation can be removed using **the principles of quantum theory**, because quantum theory naturally introduces a discontinuity in determining the dynamic state of the systems (discrete quantum state) without applying any division of the space cells. Shows that for statistical purposes, this discontinuity is equivalent to the division of the phase space of the cells having a volume equal to h^v , where h – Planck's constant ($h = 6,55 \times 10^{-27}$

erg.sek) and v – the number of degrees a free system. To emphasize that in quantum statistical theory disappears any uncertainty in determining π and S . Statistical interpretation of the Nernst's theorem: *thermodynamic state of the system at absolute zero corresponds only to a dynamic state, namely dynamic state with the lowest energy consistent with a **crystalline structure** or a **physical state** of the system.* The hypothesis that the entropy is a function of the probability π

$$S = f(\pi), \quad (\text{S})$$

associated thermodynamics, mathematics and mathematical physics. For a system of two parts to it S , π and v equations are

$$S = S_1 + S_2, \quad \pi = \pi_1 \pi_2, \quad v(\pi_1 \pi_2) = v(\pi_1) + v(\pi_2). \quad (\text{IV, 1, 2, 3})$$

If the parameters of the thermodynamic system p , V and T are selected two independent variables, for example T , V and Eq. (1, Entropy Change) was prepared

$$dQ = (\partial U / \partial T)_V dT + [(\partial U / \partial V)_T + p] dV, \quad (\text{V, VI})$$

$$dS = dQ/T = \frac{1}{T} (\partial U / \partial T)_V dT + \frac{1}{T} [(\partial U / \partial V)_T + p] dV,$$

entropy as a function of variables T , V system.

$$S = S(T, V), \quad dz = M(x, y)dx + N(x, y)dy, \quad (\text{VII, VIII})$$

Equations (V) and (VI) defined entropy – generalized function Eq. (VII), most commonly used in physics [1, 2] and even outside of it. Equation (VIII) is full differential of function with two independent variables i.e. for us equations (VII) and (VIII) – very important methodological circumstance.

1.2 Non-equilibrium thermodynamics

Important definition of **dissipative systems** (Latin disipatio – scattering, destruction) (DS): open thermodynamic systems far from thermodynamic equilibrium; exchange energy and mater (substance) with environment. DS is sustainable state occurred in non-equilibrium environment with condition for dissipation (scattering the energy that comes from outside). **Dissipative structure** (introduce by I. Prigogine [12]): dissipative system with dynamic regime in reproducible state i.e. dynamical regime is stably reproduction in the time. The state of stable reproduction can be caused of natural evolution of the system; artificial (scientific or technological requirement).

The phase transitions run in (OTS) and they are described by the thermodynamics. The extended form of the second low of I. Prigogine for (OTS) is [12]:

$$\dot{S} = \dot{S}_I + \dot{S}_E, \quad (\text{Entropy, OTS})$$

where S_I – internal entropy production in the OTS; S_E – entropy production from exchange flow energy and mater with environment. For technological system we add generalized principle of control of OTS [15]:

Control on account of information about the external effect on technological object feedback main processes (from indirect information carrier). (Control)

It is well know, that the phase transitions is classification of Ehrenfest: Phase transition of first order (see Fig. 1 solid $\leftrightarrow$ liquid $\leftrightarrow$ gas involve), because they involve a discontinuous change in density, which is the (inverse of the) first derivative of the free energy with respect to pressure; Phase transition of second order are continuous in the first derivative (the order parameter, which is the first derivative of the free energy with respect to the external field, is continuous across the transition) but exhibit discontinuity in a second derivative of the free energy.

Theoretical model of structure formation at phase transition of 1-st order in casting process is [10]

Description of solidification non-stationary temperature field is the theory of conductivity – Stefan-Schwarz problem

$$\begin{aligned}
c\rho\partial_t T &= \lambda\Delta T \quad \text{in } V = V_C \cup W_{C|M} \cup V_M \cup \Gamma, \\
T(x, y, z, t=0) &= f(x, y, z), \\
W_{C|M} : \lambda_C \partial_n T_{C|W} &= \alpha_W [T_{W|C} - T_{W|M}] \quad (x, y, z, t) = \lambda_M \partial_n T_{M|W}, \\
\Sigma : \lambda_M \partial_n T_{\Sigma|M} &= \alpha_\Sigma [T_{\Sigma|M}(x, y, z, t) - T_{Ar.M.}] \quad (1, 1 \div 8) \\
C : \begin{cases} (\lambda, \rho, c)_L & \text{at } T \geq (T_m + \Delta_m) \\ (\lambda, \rho, c + Q_m S_F)_L & \text{at } T \in (T_m + \Delta_m, T_m - \Delta_m), \\ (\lambda, \rho, c)_S & \text{at } T \leq (T_m - \Delta_m) \end{cases} M : (\lambda, \rho, c)_M, \\
S_F(T) &= \frac{1}{D\sqrt{2\pi}} e^{-\frac{1}{2}\left(\frac{T-T_{is}}{D}\right)^2} \quad \text{with condition } \int_{T_m-\Delta}^{T_m+\Delta} S_F(T) dT = 1.
\end{aligned}$$

Description of temperature field of moving overcooling zone by the next developed Stefan problem [9]

$$\begin{aligned}
\dot{T}_1 &= a_1 \partial^2 T_1 / \partial x^2 \quad \text{in } \Omega_1; \\
\dot{T} &= a \partial^2 T / \partial x^2 + R(x, t) \quad \text{in } \Omega; \\
\dot{T}_2 &= a_2 \partial^2 T_2 / \partial x^2 \quad \text{in } \Omega_2; \quad (2, 1 \div 9) \\
T(0, x) &= T^i; \\
T(t, x_i)_{|_{t=0}} &= T(t, x_i)_{|_{t=0}} = T_i, \quad i=1, 2; \\
T(t, 0) &= T^i; \quad T(t, \infty) = T^i; \\
\dot{x}_1 &= L_1 [\lambda_1 \partial T / \partial x_{|_{x=0}} - \lambda_2 \partial T / \partial x_{x=+0}], \\
\dot{x}_2 &= L_2 [\lambda_1 \partial T / \partial x_{|_{x=0}} - \lambda_2 \partial T / \partial x_{x=+0}];
\end{aligned}$$

Classical crystallization description thermodynamic and kinetic [14]

$$\begin{aligned}
\Delta T &= \Delta T_r + \Delta T_k + \Delta T_\alpha; \\
\Delta \mu &= \Delta S(T_m - T) = \Delta S \Delta T = Q_m \ln \frac{T_m}{T}; \quad (3, 1 \div 4) \\
W(n) &= -n \Delta \mu + F(n) \sigma; \\
\dot{Z}_n(\tau) &= \sum_m [f_{mn}(\tau) Z_m(\tau) - f_{nm}(\tau) Z_n(\tau)] + \\
K_n(\tau) - L_n(\tau) & \quad \text{at } \tau \in [0, t_f]
\end{aligned}$$

където for Stefan-Schwarz problem: λ, ρ, c – thermal conductivity, density, heat capacitance; $T, T_m, Q_m, T_{(CM)FE}$ – temperature field with index (C; M) of cast and mold, temperature of phase transition (melting), latent heat of melting, middle temperature of finite element; V_C is sum of volumes liquid phase (V_{CL}) inter phase surface (S_{Front}), solid phase (V_{CS}); W – work surface (cast/mold); V_M – volume of mold; Σ – external surface of mold; S_F is δ -function for approximation heat source with parameter D , which refers to the final temperature range ($T_m + \Delta, T_m - \Delta$); for Stefan problem: $R(x, t)$ is scattering function of latent heat Q_m in Ω ; $L_i = \rho / Q_m$; $a_i = \lambda_i / \rho_i c_i$ – heat conductivity number; $\lambda_i, \rho_i, c_i \quad i=1, 2$ in areas Ω_1 and Ω_2 ; in area Ω we have $a = (a_1 + a_2)/2$ $\lambda = (\lambda_1 + \lambda_2)/2$ $\rho = (\rho_1 + \rho_2)/2$ $c = (c_1 + c_2)/2$; for classical crystallization theory: ΔT_r – overcooling of the radius of curvature of the interfacial surface (dendritic peak); ΔT_k overcooling necessary to jump the atoms in interfacial surface (liquid/solid); ΔT_α overcooling of liquid phase where scattering Q_m ; $\ln(T_m/T)$ match ΔT ; $\Delta \mu = (\mu_{Liquid} - \mu_{Solid})$ – thermodynamic driving force for nucleation; W – nucleation work for nuclei which build from n (particles, atoms); σ – surface energy (tension) of nuclei n particle; F surface of nuclei build of n particles; M – maximal number of atoms (particles) in one cm^3 ($\sim 10^{39}$); Z_n and Z_m – number of complexes in the system from n or m particles; f_{mn}, f_{nm} – attachment/detachment transitions frequencies for the complexes of m - and n -particles; K_n and L_n are transition frequencies not aggregate processes introduction/removal of n -particles in/out the system; t_f – local time of solidification in volume with **suitable geometric scale**.

2. Mathematics – application in casting

Mathematical description of §1.1 is on the base of [2]. For non-equilibrium thermodynamics the necessary mathematics is much extended, because the thermodynamic system is OTS. For example Stefan-Schwarz problem eq.(1) is 3D parabolic partial differential equation with initial, boundaries conditions and with conditions for descriptions of movable solidification zone (Fig.1 e). It is fact that Stefan's problems no introduce the process of structure formation. In that reason is developed the classical Stefan task eq.(2) with possibility to obtain the size of the overcooling zone Ω where is scattering the latent heat Q_m .

For solution of Stefan problem eq.(2) is used the scattering theory [3]. This theory on the base of abstract approach of Lax and Phillips [3] where introduces the properties of two spaces D_+ outgoing and D_- incoming. These spaces allow solving the first task scattering theory: proving the existence of scattering operator. The properties of spaces D_+ and D_- are related to other tasks: presentation of core scattering; occurrence of disappearing solutions and more. The second major trend is inverse tasks which allow obtaining: information for geometric form of the obstacle: if known, the main member of the amplitude of the scattering; the maximum feature of the core of the operator of the scattering and others. The inverse problems have an extremely important practical significance. Example of inverse problem is [4].

In [5] were studied hyperbolic systemic of first order in the appearance of a limited obstacle, which border on the obstacle is set boundary condition. In too particle case the hyperbolic systemic of first order can be reduced to one equation, but in general, this is not possible. It is obtained unified approach to a broad class hyperbolic system of first order: **Problem A**: Existence of wave operators and their completeness of dissipative systems having possibly zero speed diffusion. **Problem B**: Presentation of the kernel operator dissipation for dissipative hyperbolic systems, having possibly zero speed distribution. **Problem C**: When dissipative hyperbolic systems possess or do not possess disappearing solutions? **Problem D**: Relationship between operators and vanishing outgoing and incoming spaces.

Mathematical scheme: The properties of the two spaces D_+ and D_- are

$$\begin{cases} U_0(t)D_\pm \subset D_\pm \text{ for } t > 0 \\ U_0(t)D_\pm \subset \{0\}, \text{ for } t < 0, \\ \bigcap_t U_0(t)D_\pm = \{0\}, \\ \overline{\bigcup_t U_0(t)D_\pm} = H_0. \end{cases} \quad (D_\pm)$$

Here $\{U_0(t)\}$ is group of unitary operators, acting in Hilbert space. Troubled system is described by strongly continuous semigroup of operators shrinking $\{V(t); t \geq 0\}$, acting in Hilbert space $H \subset H_0$. For construction of the abstract approach is supposed that the property is satisfied H_1, H_2, H_3 and H_4

$$\begin{aligned}
a) & A_j^*(x) = A_j(x), \\
b) & \text{has a number } R_0 > 0 \text{ so that } R^n \setminus \Omega \subset \{x; \|x\| < R_0\}, \\
& \text{and } A_j(x) - A_j^0 = B(x) = 0 \text{ for } \|x\| \geq R_0, \\
c) & \text{the matrices } A(\xi) = \sum_j A_j^0 \xi_j
\end{aligned} \quad H_1$$

it has values at a constant multiplicity of $\xi = (\xi_1, \dots, \xi_n) \in R^n \setminus \{0\}$.

$$\begin{cases} \text{the matrices } A(x, \nu(x)) = \sum_j A_j(x) \nu_j(x), \\ \text{it has permanent rank for } x \in \partial\Omega. \end{cases} \quad H_2$$

- a) $B^*(x) + B(x) - \sum_j \partial_{x_j} A_j(x) \leq 0$ for $x \in \overline{\Omega}$,
b) $(u, A(x, v(x))u) \leq 0$ for $u \in \text{Ker} \Lambda(x)$, $x \in \partial\Omega$, H_3
c) the linear subspace $\text{Ker} \Lambda(x)$ of C^d
is the maximum subspace of C^d ,
for which is in force the properties b).

G is dissipative operator, generated semigroup and coercive. The norm in space H we will note with $\| \cdot \|$. Domain $\mathcal{D}(G)$ of the operator G is the closing on the norm $\|f\| + \|Gf\|$. It is obtain the equality

$$G\varphi = \lim_{t \rightarrow 0} \frac{1}{t} (V(t)\varphi - \varphi)$$

where $\varphi \in \mathcal{D}(G)$; V(t) is shrinkage operator if $\|V(t)f\| \leq \|f\|$ for $f \in H$. Denote H_b a linear subspace in H caused by eigenvalues vectors of G with corresponding eigenvalues on the imaginary axis. Let $H_b^\perp$ is orthogonal addition of H_b in H. In force assessment

$$\| \partial_x f \| \leq C (\| Gf \| + \| f \|) \text{ for } \mathcal{D}(G) \cap H_b^\perp. \quad H_4$$

Wave operator is define

$$Wf = \lim_{t \rightarrow \infty} U_0(-t)V(t)f \text{ for } f \in H_b^\perp. \quad (\text{Wave operator})$$

The space D_+ is outgoing and the space (D_-) is incoming) if $f \in H_{ac}(G_0)$ and $R(f)(s, \omega) = 0$ for $s < 0$ ($s > 0$).

Main idea: to seek representation of the distributions $\tilde{S}_{\mu\nu}^{jk}(s, \sigma, \theta, \omega)$, which define the core of operator scattering in accordance to $S(s, \sigma, \theta, \omega) = \sum_{j=1}^{m/2} \sum_{k=1}^{m/2} \{ \tilde{S}_{\mu\nu}^{jk}(s, \sigma, \theta, \omega) \}_{\mu, \nu=1}^d$. And finite result in [5] is the core

$$\tilde{S}(s, \sigma, \theta, \omega) = \delta(s - \sigma) \delta(\theta - \omega) I + S(s - \sigma, \theta, \omega). \quad (\text{Ker})$$

The mathematics of this paragraph is used to solved the eq.(2) [9] in theoretical model for structure formation in [10].

3. Mathematical physics in casting

The nature of the casting process is the creation of a polycrystalline structure – the working properties of the castings. It is therefore required to use fundamental new scientific results. Elegant example is work [6] “at the end of 1907 Einstein holds in his hands the physical principle and the main consequences of the future theory; however, it takes him eight years - and the involvement of mathematicians Grossman and Hilbert - to reach its precise formulation”. If we follow [6] based on historical experience of the development of science in the 20-th century we have methodological basis: mathematics and physics; Mathematics and Mathematical Physics; computational physics. In that subject from [7] and [8] we can conclude – industry need pure mathematics.

The Fig. 1 is picture for micro-foundry: mathematical description in solidification zone introduce not only numerically of phase transition like area, but and descript and polycrystalline structure formation with maximum details. The first is mathematical theory of scattering for introduction driving force of crystallization [9]. Second is description polycrystalline structure formation by quantum mechanics: the equations of the molecular mechanics

$$\ddot{m}_i \ddot{r}_i = f_i \quad f_i = \partial U / \partial r_i, \quad (\text{MM})$$

$$-\hbar^2 \nabla^2 \psi(r) / 2m + V(r) = E \psi(r) \quad (\text{Schrödinger equation, 1, 2})$$

$$i\hbar \partial \psi(r, t) / \partial t = -\hbar^2 / 2m \partial \psi(r, r) / \partial r^2$$

where m , r and f_i – mass, coordinates and acting on atomic force derived from potential energy $U(r^N)$, where $r^N = (r_1, r_2, \dots, r_N)$ introduced full set of $3N$ coordinates of atoms; Planck's constant –

$\hbar$; ψ - wave function, m – mass of the electron. The potential energy can introduce: interaction between not connection or connected atoms; the atoms and electrons building the crystal lattice and behavior of electrons are describes with Schrödinger's equation equation.

For diffusion process is use task only in one dimensional case $\partial C / \partial t = D \partial^2 C / \partial x^2$, where C – concentration, x – coordinate, D – diffusion coefficient [11]. This approach is convenient to applied, because we have growth nuclei with different size i.e. different scales.

3. Conclusion

Micro-foundry is need of innovations of fundamental results from mathematics and mathematical physics. This can be done by creating temporary teams of scientists from the Bulgarian Academy of Sciences, Universities and user micro-foundry with the support of industry branch in this case machine-building.

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LOCAL POST WELD HEAT TREATMENT OF WELDED STEEL PIPES

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Abstract: In this paper, local post weld heat treatment (LPWHT) effects on the metallurgical and mechanical properties of high frequency electric resistance welded (HFERW) steel pipes were investigated. Local post weld heat treatment (LPWHT) of the welded joint was carried out by an induction heating device which selectively heat only the weld area from the outside surface of the welded steel pipes. Optical microscopy, tensile testing, Charpy-V-notch testing, hardness testing and flattening testing were used to evaluate local post weld heat treatment (LPWHT) effects on the metallurgical and mechanical properties of high frequency electric resistance welded (HFERW) steel pipes.

KEYWORDS: HEAT TREATMENT, MECHANICAL PROPERTIES, TECHNOLOGICAL PROPERTIES.

1. Introduction

Welding is a complex process and the quality of a weld is a function of interaction of significant number of variables together with the microstructural changes attendant upon welding. High-frequency electric resistance welding (HFERW) process is one of the most extensively methods for production of high quality longitudinal seam welded carbon steel pipes suitable for line pipe, casing, tubing and for other constructive uses. In this process, hot rolled strip is gradually formed into round shape through roll-forming stands, and its edges are joined by a combination of localized high-frequency electric resistance heating and forge pressure, as schematically illustrated in figure 1 [1, 2]. Forge pressure imposes significant plastic deformation to the welded joint and this represents a peculiarity of such welding process. Most of the high frequency electric resistance welded steel pipes are produced according to API Standard [3] plus client supplementary requirements.

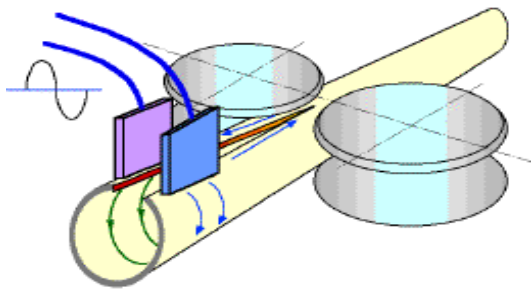


Fig.1 Schematic illustration of HFERW

The high-frequency current applied to the strip edges through sliding contacts, concentrated on the surface layer of the strip edges due to the "skin" and "proximity" effects, generate joule heat and the hot "Vee" converge edges are forged together in the weld squeeze rolls and a forge type weld is achieved [4]. The HF weld is a true forge weld in that no filler metal is added and, if done properly, no molten or oxidized metal is left on the bond line. Figure 2 shows that all of the molten thin layer and metal oxides and other inclusions are squeezed out of the weld toward the outside and inside surfaces as the edges pass between the weld rolls.

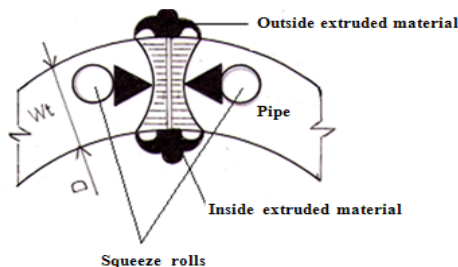


Fig.2 High frequency forge type weld

The material extruded on the inside and outside weld surfaces, usually removed mechanically while still hot and forms a typical

HFERW weld with narrow bond line (BL) or fusion line (FL) and associated heat affected zone (HAZ) is formed. The normal HFERW weld area is shown in figure 3 [4]. Note that the heat affected zone is shaped like an hourglass.

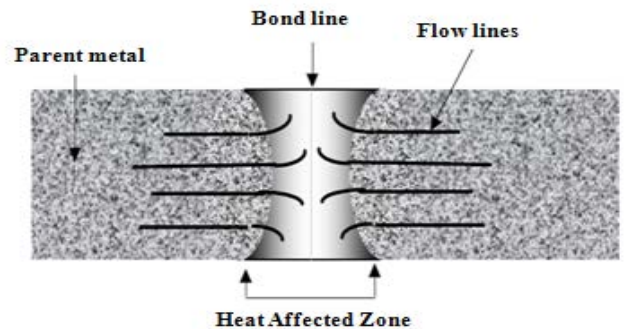


Fig.3 High frequency electric resistance weld area

The plastic deformation which is realized under the action pressure of the squeezing rolls caused metal flows of the hot rolled coils towards the outside and inside surfaces of the bond line. This flow pattern consists of streaks and striations. The orientation of this pattern, with respect to the new surface, indicates the direction of metal flow lines (fibres), respectively flow angles during plastic deformation [5].

The high frequency electric resistance welded joint is subjected to a local post weld heat treatment (LPWHT) in-line by the inductive heating, followed by air cooling. The aims of this heat treatment is to produce certain microstructure and desired mechanical properties (hardness, yield strength, ultimate tensile strength, toughness, ductility etc.). During local post weld heat treatment (LPWHT) of the welded joint, depending on the temperature range and the cooling rate, metallurgical changes takes place and these changes covers the properties of the welded joint and the performance of welded pipes in service [6, 7, 8, 9, 10].

Therefore the objective of this paper was to investigate local post weld heat treatment (LPWHT) effects on the mechanical and metallurgical properties of high frequency electric resistance welded steel pipes.

2. Experimental procedure

High-frequency electric resistance welding (HFERW) was conducted using an L-16 inch, pipe mill Newco IMK-Ferizaj, Kosova. Newco IMK-Ferizaj-Kosova produces high frequency electric resistance longitudinal seam welded steel pipes with Ø114.3-406.4mm diameter and 3.2-12.7mm wall thickness. In this paper Ø 406.4 x 6.35mm welded pipes were investigated. Pipes Ø 406.4 x 6.35 mm were fabricated from high strength steel coils X52 according to API [3, 11] standard, using the pipe mill equipped with a contact type high frequency electric resistance welding machine-Thermatool. The frequency of current was 400kHz.

Chemical composition and mechanical properties of the used steel are given in Table 1 and 2.

Table 1: Chemical composition of steel API Grade X52

Steel coils	Chemical composition [wt-%]						
	C	Mn	Si	P	S	Al	V
API grade X52	0.140	1.25	0.23	0.018	0.015	0.020	0.041

Table 2: Mechanical properties of used steel coils X52

Steel coils	Re	Rm	Re/Rm	A ₂ ''	KV
					ISO-V-0°C
	MPa			%	J
API grade X52	399	497	0.802	32.5	121

The induction local post weld heat treatment (LPWHT) of the welded joint (bond line-BL and heat affected zone-HAZ) of the produced pipes were performed in line by inductive heating device, figure 4 which is located in the continuation of the production line L-16 inch and makes only selective heating of the welded joint, figure 4, in three zones:

- in the first zone, heating at the temperature 740-760°C,
- in the second zone, heating at the temperature 840-860°C,
- in the third zone, heating at the temperature 900-920°C.

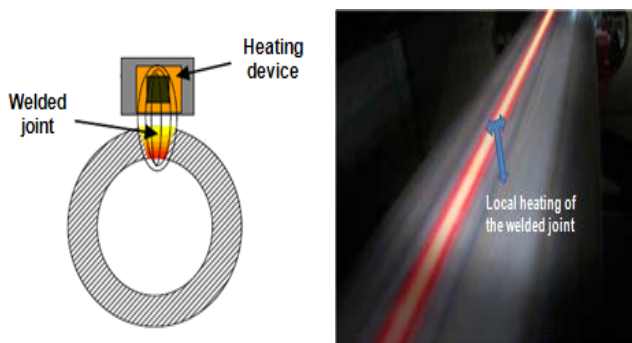


Fig.4 In line local post weld heat treatment of HFERW of welded pipes

Whether or not appropriate local post weld heat treatment (LPWHT) were performed, in this presentation confirmed by:

- tensile testing,
- impact toughness testing,
- hardness testing,
- flattening testing
- metallographic analysis.

Specimens were obtained before (as welded) and after (as reheated) local post weld heat treatment (LPWHT), according to the illustration in figure 5a and 5b.

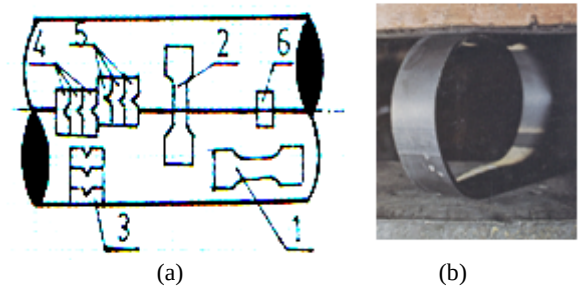


Fig.5 Sketch illustrating the specimens used for mechanical testing (a) and pipe ring for flattening testing (b)

Tensile testing were carried out on the base metal (BM), specimen-1 and on the welded joint (WJ), specimen-2, figure 5a.

Impact toughness testing were carried out at the temperature (0°C), by Charpy V-notch (CVN) specimens on the base metal (BM), specimen-3, on the bond line (BL), specimen-4 and on the heat affected zone (HAZ), specimens-3, figure 5a.

Hardness testing across the welded joint, figure 6 were carried out using Vickers (HV30/15) method, specimen-6, figure 5a.

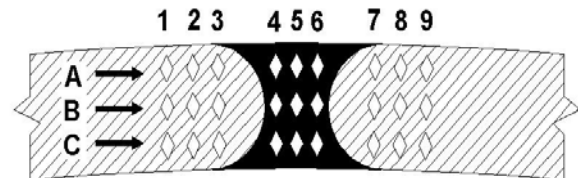


Fig.6 Hardness (HV30/15) measurement positions

Pipe rings (15-20 cm long) were extracted from several pipes for use as the specimens for flattening test, figure 5b.

In order to determine metallographic macro and micro analysis by optical light microscope (NEOPHOT), the specimens from hardness testing or from flattening testing were prepared by standard metallographic techniques that includes grinding, polishing and etching with suitable etchant (2% nital).

3. Results and discussion

Tensile specimens before and after local post weld heat treatment (PWHT) are subjected to the tensile testing on the universal machine MOHR-FEDERHAFF-LOSENHAUSEN, and obtained results are shown in figure 7.

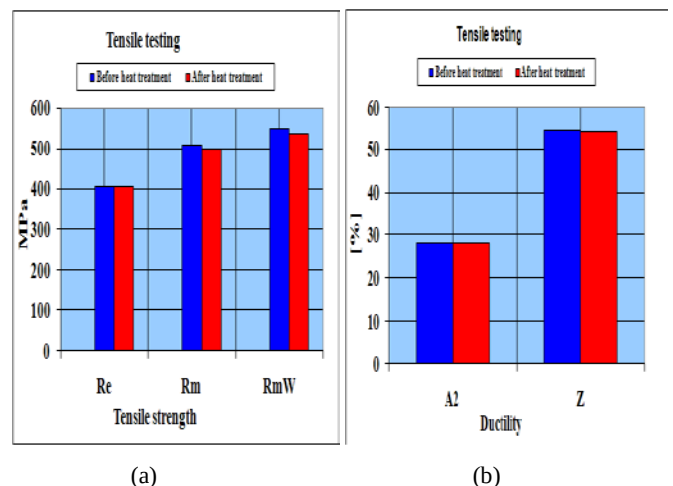


Fig.7 Tensile testing characteristics

From the tensile testing characteristics in this case, only ultimate tensile strength of the welded joint (Rm_W), specimen 2,

slightly decreased after local heat treatment, but obtained values are above the minimum required values by the API standard. It should be noted that in all cases fracture occurs in the base metal, far from the welded joint, figure 8.

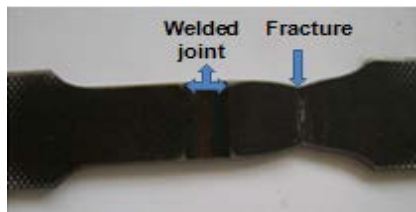


Fig.8 Tensile testing specimen after tensile testing

Results from the impact toughness testing in the Charpy pendulum, MOHR- FEDERHAFF-LOSSENHAUSEN, are shown in figure 9.

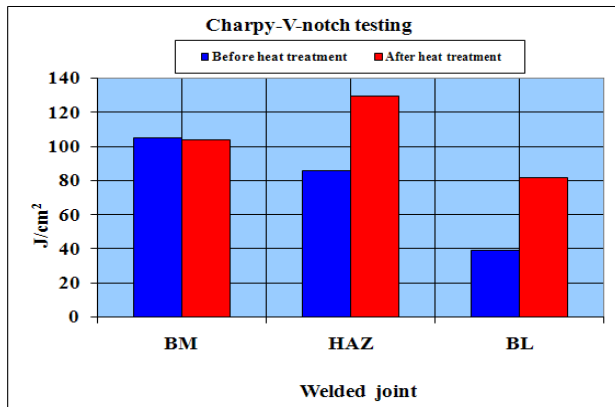


Fig.8. Impact toughness characteristics

Average value of three test results of bond line (BL-specimen-4) and heat affected zone (HAZ-specimen-5) increased as a result of the local post weld heat treatment (PWHT) of the welded joint, figure 8.

Hardness measurements across the welded joint were carried out using (HV30/15) method according to the schematic illustration in figure 6 and the obtained results are shown in figure 9. From the hardness test results, it can be deduced that hardness of the welded joint, particularly the hardness of the heat affected zone (HAZ) and hardness of the bond line (BL), after local post weld heat treatment (PWHT) decreases significantly, figure 9. After local post weld heat treatment (LPWHT) hardness distribution across the welded joint is uniform, figure 9.

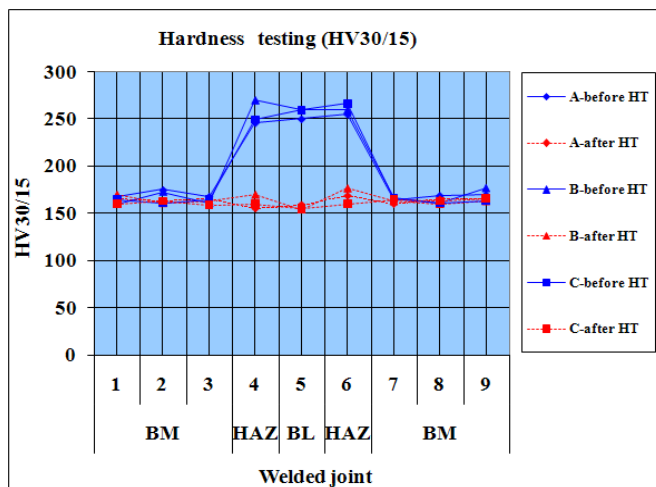


Fig.9 Hardness (HV30/15) testing results

Fracture surfaces from the Charpy-V-notch testing are shown in figure 10, separately from base metal (BM), heat affected zone (HAZ) and bond line (BL), before and after local heat treatment.

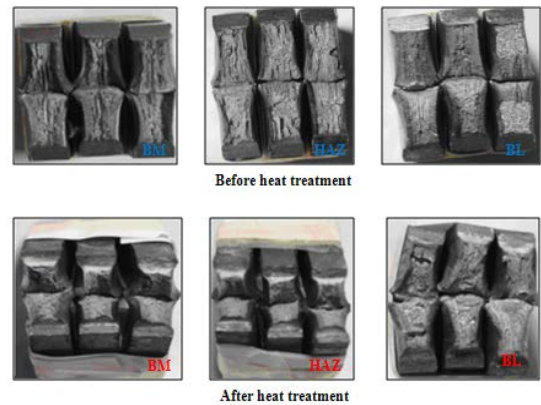


Fig.10 Fracture surfaces from Charpy-V-notch (CVN) testing

The test specimens (rings for flattening testing) were flattened in a hydraulic press, DIHNAFORIN PS 2043, at room temperature between two parallel plates with the weld line located 90° and 0° to the applied force, figure 11.

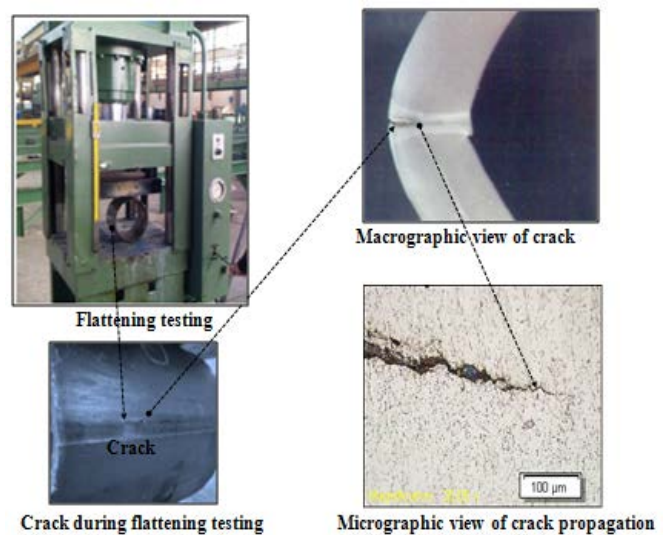


Fig.11 Flattening testing and view of crack

In all Specimens before local post weld heat treatment (LPWHT) were occurred cracks or breaks within or in the vicinity of welded joint, which path of propagation is different within welded joint and these results indicates low ductility of welded joint. Some fracture surfaces from the flattening testing are shown in figure 12.

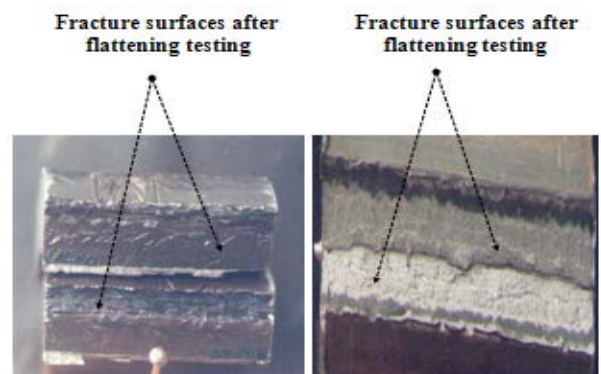


Fig.9 Fracture surfaces from flattening testing

During flattening testing, after local post weld heat treatment (LPWHT) is no occurrence of any cracks or breaks in the weld area in the position (90° and 0°) until the height specified according to API.

4. Conclusion

The following conclusions can be drawn from the present study:

Welded joint of high frequency electric resistance welding (HFERW) steel pipes, undergoes considerable metallurgical changes during local post weld heat treatment (PWHT). Metallurgical changes that occur in the forge weld area during local post weld heat treatment (LPWHT) are a result of the temperature range and cooling rate. These metallurgical changes refine and unify the final microstructure across the welded joint and improve mechanical properties of the welded joint, especially ductility and impact toughness.

Local post weld heat treatment (LPWHT) of welded pipes is an important constitutive part of the steel pipes production line and through metallurgical changes covers the properties of the welded joint and the performance of welded pipes in service.

Local post weld heat treatment (LPWHT) is recommended after high frequency electric resistance welding (HFERW) to refine and unify grain size structure and improve mechanical properties of the steel pipes welded joint. The mechanical properties that develop as a function of local post weld heat treatment (LPWHT) have been correlated to the microstructural evolution.

The best parameters for evaluation of the effects of local post weld heat treatment (LPWHT) are: toughness values, hardness values and occurrence of cracks and breaks in the welded joint, so Charpy-V-notch testing, hardness testing and flattening testing, accompanied with standard metallographic analysis are valuable and necessary testing in the production of welded steel pipes by high frequency electric resistance welding (HFERW).

The results of this investigation have demonstrated that the X52 steel grade meets all the accepted requirements according to API standard after local post weld heat treatment (LPWHT).

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FRACTOGRAPHIC ANALYSIS OF WELDED JOINT SURFACES

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Abstract: In this paper are presented some macrofractographic and microfractographic analysis of welded joint fracture surfaces, respectively welded seam (W), heat affected zone (HAZ) and base metal (BM), of spiral and longitudinal steel welded pipes after conducting the laboratory destructive testing. Destructive testing are conducted to assess the quality of the steel welded pipes and besides the numerical results, the fracture surface of the tested samples offers additional information, very important for assessing the quality of steel welded pipes. Given this, in the paper are treated the macrofractographic and microfractographic analysis (LOM-Light Optical Microscopy and Scanning Microscopy-SEM) of the fracture surfaces of samples which are fractured after destructive testing. The morphology of the fracture surfaces is compared with the numerical results and it concludes that there is a direct correlation between the obtained results and deformation that causes fracture, providing thus additional information for assessing the quality of the welded pipes.

KEYWORDS: MACROFRACTOGRAPHY, MICROFRACTOGRAPHY, FRACTURE SURFACE, DEFORMATION.

1. Introduction

A fracture is the separation of an object or material into two, or more, pieces under the action of stresses and fractography can be defined as the science which deals with the description, analysis and interpretation of fracture surface morphologies (fracture topographies) and links them to the causative stresses, mechanisms and subsequent evolution of the fractures. Fractography can be classified according to the field (scale) of observation into [1]:

- macrofractography, dealing with features which can be studied visually using the naked eye or hand lens, and
- microfractography, dealing with features which can be studied at low magnification using Light Optical Microscopes (LOM), or at high magnification with Scanning or Transmission Electron Microscopes (SEM or TEM). Energy-dispersive X-ray (EDS) spectroscopy, used in conjunction with the SEM or TEM, is a very practical tool for elemental chemical analysis [1, 2, 3].

Fracture of materials depends upon many parameters like the state of stress, strain rate, the defect size, crystal structure, chemical homogeneity, grain size, grain boundary, formation of twins and slip plane, temperature and environmental conditions [4].

Based on the ability of a material to experience plastic deformation, for engineering materials, two fracture modes are possible: ductile and brittle [5]. Ductile fractures typically exhibit substantial plastic deformation with high energy absorption before fracture. On the other hand, brittle fractures display little or no macroscopically visible plastic deformation and require less energy to form. Fractured surfaces display both macroscopic and microscopic features.

Ductile fractures exhibit certain characteristic macroscopic features:

- a relatively large amount of plastic deformation precedes the fracture,
- shear lips are usually observed at the fracture termination areas,
- the fracture surface may appear to be fibrous or may have a matte or silky texture, depending on the material,
- the cross section at the fracture is usually reduced by necking,
- crack growth is slow.

Macroscopically, brittle fractures are characterized by the following:

- little or no visible plastic deformation precedes the fracture,
- the fracture is generally flat and perpendicular to the surface of the component,

- the fracture may appear granular or crystalline and is often highly reflective to light. Facets may also be observed, particularly in coarse-grain steels,

- herringbone (chevron) patterns may be present

- cracks grow rapidly, often accompanied by a loud noise.

On a microscopic scale, ductile fracture in metals displays a dimpled surface appearance created by microvoid coalescence and transgranular separation.

Microscopically, brittle fractures have the following characteristics:

- transgranular cleavage or quasi-cleavage,

- intergranular separation,

- features on transgranular facets, such as river marks, herringbone patterns, or tongues.

2. Experimental procedure

Spiral line pipes Ø813x12mm were fabricated using high strength steel coils X65 according to API 5L (American Petroleum Institute) standard, which chemical composition and mechanical properties are given in table 1 and 2, according to the Certificate of Quality. Spiral line pipes Ø813x12mm were welded in two-stage process by double sided Submerged Arc Welding (SAW) through an "X" groove configuration, according to the BLOHM+VOSS, figure 1.

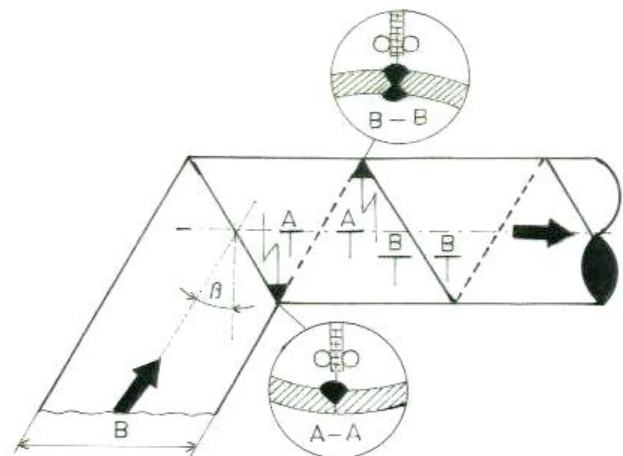


Fig.1 Double sided submerged arc welding (SAW)

Table 1: Chemical composition of steel API Grade X65

Steel coils	Chemical composition [wt-%]					
	C	Mn	Si	P	S	V
API grade X65	0.09	1.31	0.43	0.018	0.005	0.048

Table 2: Mechanical properties of used steel API Grade X65

Steel coils	Re	Rm	Re/Rm	A ₂ ”	KV
					ISO-V-0°C
	MPa			%	J
API grade X65	549	649	0.845	23.6	126

Charpy V-Notch (CVN) specimens were cut out from the double sided weld metal, perpendicular to the welding direction, as shown in figure 2.

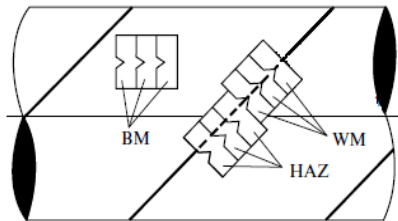


Fig.2 Schematic representation of the Charpy V-Notched specimens

The standard CVN specimens (10x10x55mm) with V groove were cut from base metal (BM), heat affected zone (HAZ) and weld metal (WM) and tested at (-20°C), (0°C) and (20°C) by use of the MOHR-FEDERHAFF LOSSENHAUSEN pendulum.

During Charpy impact testing, usually three key parameters are typically determined:

- I-impact toughness,
- II-presence of shear and brittle fracture area and
- III- lateral extension.

A methodology for measurements of the presence of shear (ductile) fracture area and brittle fracture area on the total fractured surface of a CVN tested specimens is shown in figure 3. The specimen cross-section after fracturing is divided into equal millimetric units and then by counting of the relevant squares into the entire cross-section, using Autocad software, determines percent of shear fracture (ductile) area and percent of brittle fracture area.

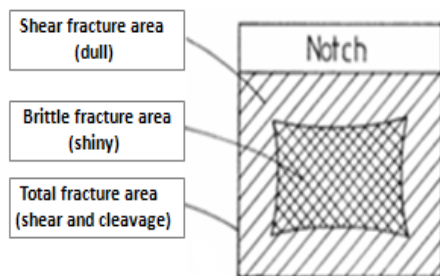


Fig.3 Shear and brittle fracture surface

In order to obtain a more complete assessment of the fracture surfaces of Charpy V-notched specimens, lateral extension were measured [6], in the height direction (h_1-h_0) and in the width directions (a_1-a_0), figure 4, where:

- h_0 , a_0 (mm)-initial specimen height and width, before fracture and

- h_1 , a_1 (mm)-maximum specimen height and width after fracture.

According to the measured dimensions before and after fracture, relative strain in the height directions (ϵ_h) and relative strain in the width directions (ϵ_a) were calculated:

$$\epsilon_h = \frac{h_1 - h_0}{h_0} \times 100 [\%] \dots \dots \dots (1)$$

$$\epsilon_a = \frac{a_1 - a_0}{a_0} \times 100 [\%] \dots \dots \dots (2)$$

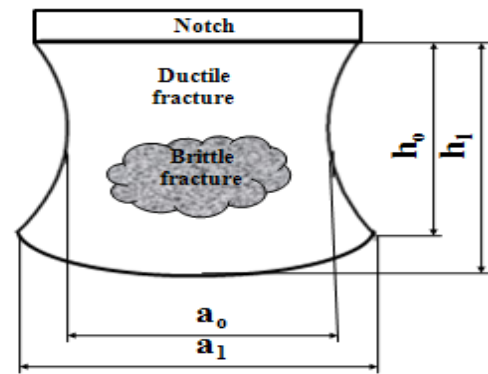


Fig.4 CVN Specimens cross section after fracture

All fractured surfaces of CVN specimens after testing were examined visually with the unaided eye, at low magnification using an Light Optical microscope (LOM-NEOPOT 21) and at high magnification using Scanning Electron Microscope (SEM-Leo 1530).

3. Results and discussion

3.1 Impact toughness

Impact toughness results of the Charpy-V Notch specimens are presented in figure 4. These values represents average of three specimens measurements at different temperatures (-20°C), (0°C) and (20°C).

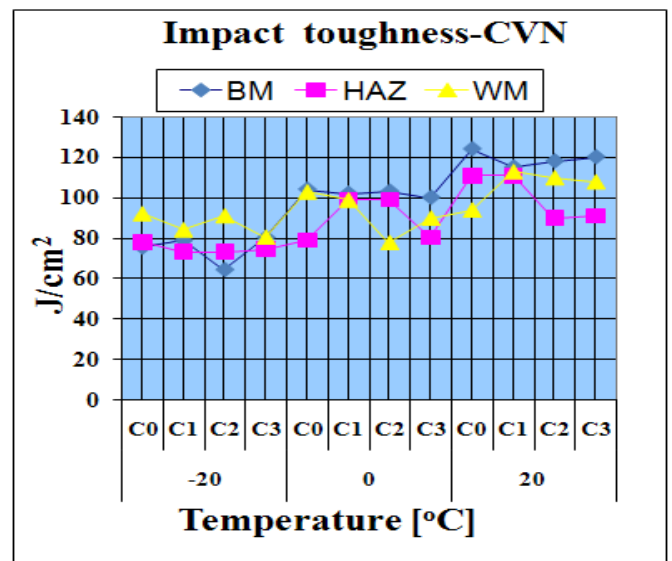


Fig.5 Impact toughness (CVN)

It can be seen from figure 5 that the base metal (BM) has the highest absorbed energy, followed by the weld metal (WM) and coarse grained heat affected zone (CG HAZ). Each data point represents the average values of three samples and the test results revealed a general decreasing tendency with decreasing of the temperature.

Visual analysis of the Charpy V-notch fracture surfaces clearly shows differences between base metal (BM), heat affected zone (HAZ) and weld metal (WM), figure 6 [7].

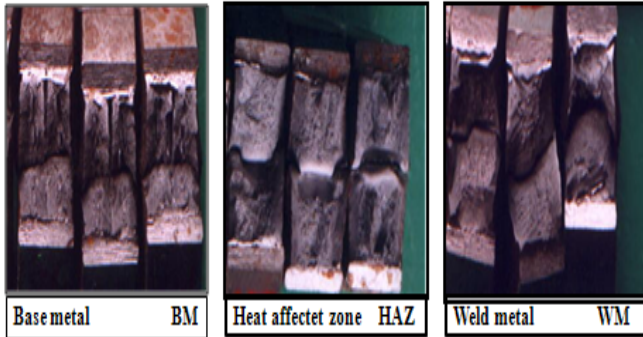


Fig.6 Typical fracture surfaces of BM, HAZ and WM

From the visual analysis, figure 5, there is notable evidence of plastic deformation in the fracture surfaces in all tested specimens of base metal (BM), heat affected zone (HAZ) and weld metal (WM). Post-fracture visual analysis in all fractured surfaces shows presence of shear lips which are located at the opposite sides of the specimens width, figure 7. Presence of shear lips is also a characteristic of ductile fracture.

Macroscopic view of the fracture surface of the Charpy V-Notch specimen of BM, specimen marked as C₂, with lowest impact energy ($Kv=64.2J/cm^2$), figure 4, tested at ($-20^{\circ}C$) is presented in figure 7.

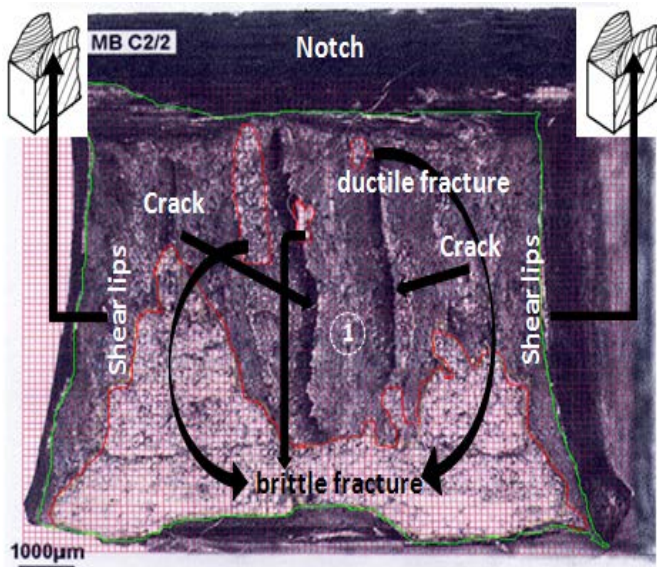
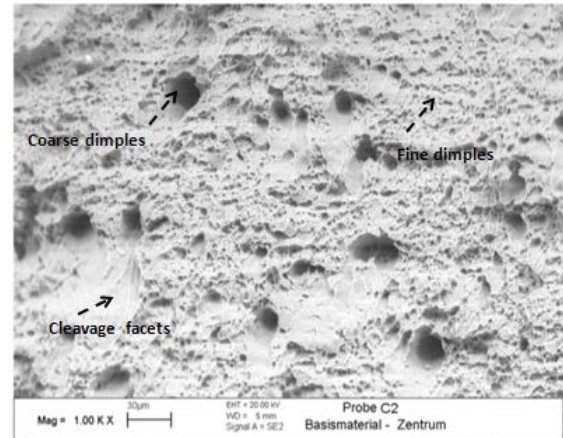


Fig.7 Macroscopic view of the fracture surface of BM

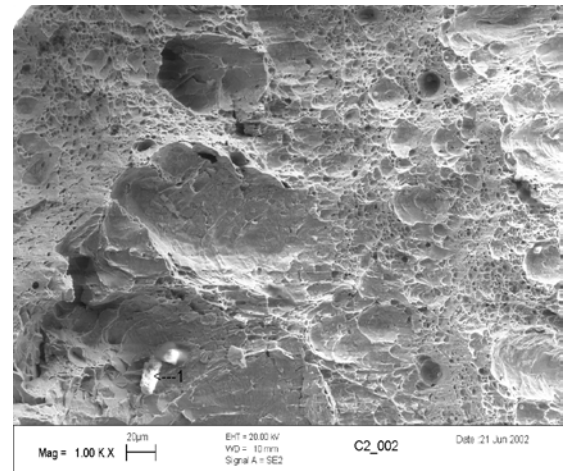
In general, all fracture surfaces of the base metal (BM), heat affected zone (HAZ) and weld metal (WM) comprises mainly of a large ductile region and small amounts of brittle area. The fracture surface of base metal (BM) is fibrous and its color is dull. Fracture surfaces of the weld metal (WM) and heat affected zone (HAZ) are also fibrous with a little presence of bright shiny color. In the case of base metal (BM) with lowest impact energy ($Kv=64.2J/cm^2$), specimen marked with C₂ and tested at ($-20^{\circ}C$), brittle area appears flat and shiny, figure 7.

Fracture surfaces of some specimens of base metal (BM) shows some cracks like separations perpendicular to the axis of the V-notch, figure 6 and 7. These cracks are referred to as delamination [7].

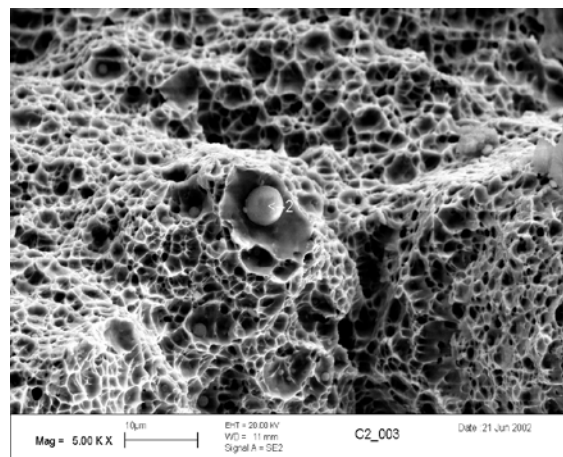
The scanning electron micro-fractography (SEM) of the central portion of the base metal (BM) specimen, marked with circle 1, in figure 8, reveals numerous coarse and fine concave equiaxed dimples, characteristic of a ductile area. Partially in this micro-fractography (SEM) exist some cleavage facets, characteristic of brittle fracture, figure 8(a), while in the rest of fracture surface dominates ductile fracture, figure 8(b, c). Microanalysis (EDS-EPMA), figure 9, of precipitation marked with number 2, figure 8(c), besides presence of other elements shows also presence of Mn, probably in form of the sulfides, which are characteristic for ductile fracture.



(a)



(b)



(c)

Fig.8 SEM micro-fractography of fracture surfaces

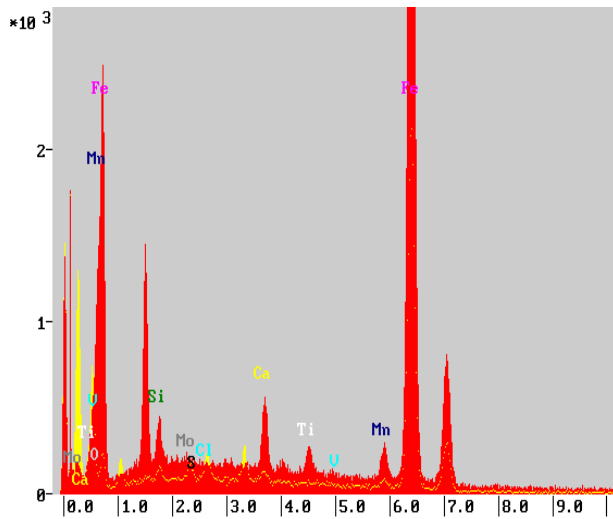


Fig.9 Spectrum from the microanalysis (EDS-EPMA)

3.2 Presence of shear and brittle fracture area

The presence of shear or ductile and brittle fracture area of CVN tested specimen in (-20°C) is shown in table 3. These values represent average of three specimens measurements.

Table 3: Presence of ductile and brittle fracture area

Steel coils	Specimens	Presence of ductile and brittle fracture area [%]					
		[BM]		[HAZ]		[WM]	
		Temperature [-20°C]					
		Ductile	Brittle	Ductile	Brittle	Ductile	Brittle
X65	C ₀	79.6	20.4	90.2	9.8	92.3	7.7
	C ₁	82.7	17.3	88.7	11.3	94.7	5.3
	C ₂	70.9	29.1	92.1	7.9	95.1	4.9
	C ₃	89.4	10.6	90.5	9.5	93.3	6.7

Table 3 shows that shear or ductile fracture dominated in all cases. The largest percentage (29.1%) of brittle fracture is only in the sample marked with C₂, which is in accordance with the numerical value of the impact toughness, given in figure 5.

3.3 Lateral extension

Lateral extension in height (ϵ_h) and in width (ϵ_a) determined by formula (1) and (2), respectively relative strain in height and in width are presented in table 4. Results of lateral extension shows that with the increasing of the absorbed energy, lateral in height (ϵ_h) and in width extension (ϵ_a) also increased. It must be noticed that the results of lateral extension not used for practical needs because their determination is difficult and is not standard method. Results of lateral extension only certify the preliminary obtained values of absorbed energy and percentage of shear and brittle fracture area.

Table 4: Relative strain of height (ϵ_h) and width (ϵ_a)

Steel coils	Specimens	Relative strain %					
		[BM]		[HAZ]		[WM]	
		ϵ_h	ϵ_a	ϵ_h	ϵ_a	ϵ_h	ϵ_a
X65	C ₀	27.1	25.2	22.8	20.1	26.2	24.2
	C ₁	26.3	24.1	21.9	18.1	24.5	20.2
	C ₂	23.6	19.1	23.9	18.2	24.2	21.9
	C ₃	24.3	21.3	24.3	20.6	23.7	20.7

4. Conclusion

On the basis of the results presented in this work, it is possible to draw the following conclusions:

-Impact toughness values accompanied with visual analysis of fractured surfaces of CVN specimens can provide additional information for quality evaluation of the welded joint, respectively base metal (BM), heat affected zone (HAZ) and weld metal (WM).

-The presence of shear or ductile and brittle fracture area in all cases is in accordance with numerical value of the impact toughness obtained during testing.

-Lateral extension measurements with determination of the percentage of ductile and brittle fracture in the total fracture area are additional parameters and facilitate the assessment of the quality of the welded joint, respectively base metal (BM), heat affected zone (HAZ) and weld metal (WM).

-Microscopic analysis with Light Optical Microscopy (LOM) and Scanning Electron Microscopy (SEM) can provide very important additional data for qualitative and quantitative assessment of fractured surfaces and mechanisms of fracture of welded joint, respectively base metal (BM), heat affected zone (HAZ) and weld metal (WM).

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COMPARATIVE ANALYSIS OF STATIC AND DYNAMIC ELASTIC MODULUS OF POLYMER CONCRETE COMPOSITES

СРАВНИТЕЛЕН АНАЛИЗ ЗА СТАТИЧНИЯ И ДИНАМИЧНИЯ МОДУЛ НА ЕЛАСТИЧНОСТ ПРИ ПОЛИМЕРБЕТОННИ КОМПОЗИТИ

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Abstract: The article presents a comparative analysis of the quantitative values obtained from the experimental tests, of the static and dynamic modules of elasticity (Young's modulus) of gamma polymer-concrete composites. The same will be used as constructive material for parts and body elements. Standard test methods are applied. The values of the elastic parameters of the tested polymer-concrete composites are necessary as input data for the various engineering analysis softwares.

Keywords: POLYMER CONCRETE COMPOSITES, DYNAMIC ELASTIC PROPERTIES, COMPARATIVE ANALYSIS, DYNAMIC MODULES.

1. Introduction

For the quantitative assessment of the elastic properties of the actual materials, the so called elastic properties are applied, which are determined by experiment. They are of important practical significance for calculating the elements and structures of strength and hardness, by the various software products for engineering analysis.

The elastic properties include the elasticity modulus E (Young's modulus), the modulus of tangential elastic deformation G (Culon modulus), Poisson's ratio μ , and the modulus of comprehensive pressure K . They are interrelated; where two of them are independent, and thus assumed as basic. Most often in practice, the elasticity modulus E and the shear modulus G are determined experimentally, while Poisson's ratio μ and the modulus of comprehensive pressure K are calculated on their basis, by well known mechanical formulae [1].

The elastic properties have a certain physical meaning, and for the traditional materials they are assumed as constants. For polymers and polymer composites it is found experimentally that the values of the elastic properties determined in the cases of static loading differ considerably from the values determined by the dynamic methods. This difference for some composites can be significant, [5].

Subject of the study of this article are 15 thermoreactive, quasi-isotropic, viscoelastic polyester polymer concrete composites, made in the Laboratory for testing and studying metal-cutting machines, at Technical University of Sofia, Plovdiv Branch [6]. The same will be used as structural material for bodies and body parts of production machines.

Subject of study of this work are: the elastic properties of the range of polymer concrete composites.

2. Theoretic prerequisites

It is well known the method for determining experimentally the static Young's modulus on the basis of the standard ASTM C 580 - 02 (2012), [4]. The core of the method consists of testing by three-point bending of the experimental samples, where the cross sections of the sample "girder" are characterized by normal tensions of the bending moment, Fig. 1. Loading the samples with focused static force, we measure the sagging f at the loading point.

Within the limits of proportionality, where Hooke's law is valid, we can properly write the following:

$$f = \frac{Pl^3}{48EJ_y} \quad (1)$$

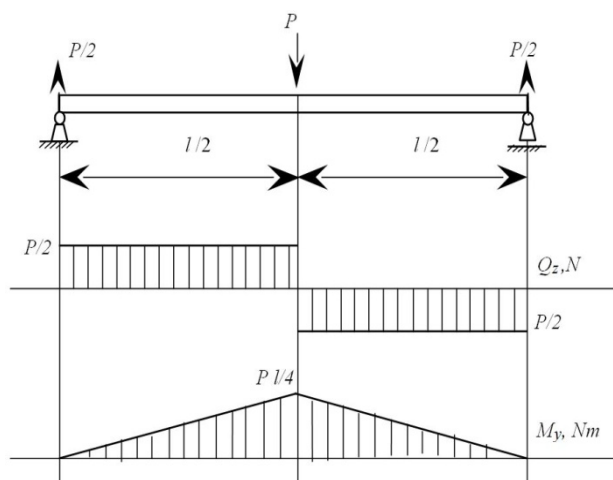


Fig. 1. Diagrams for bending

where:

P - is a focused static force;

EJ_y - hardness of bending;

E - linear deformation modulus;

J_y - moment of inertia versus the central inertia axis y ;

l - distance between the supports.

From (1) we express the force P :

$$P = \frac{48EJ_y}{l^3} \cdot f, \quad (2)$$

The inertia moment for rectangular section is:

$$J_y = \frac{b \cdot h^3}{12} \quad (3)$$

where:

b - is the width of the sample section;

h - height of the sample section.

We can write the following after substituting of (3) in (1) and (2):

$$f = \frac{Pl^3}{48Ebh^3} \quad (4)$$

$$P = \frac{48Ebh^3}{l^3} \cdot f \quad (5)$$

The functional relation between the loading force P and sagging (deformation) f of the sample body looks as shown on Fig. 2.

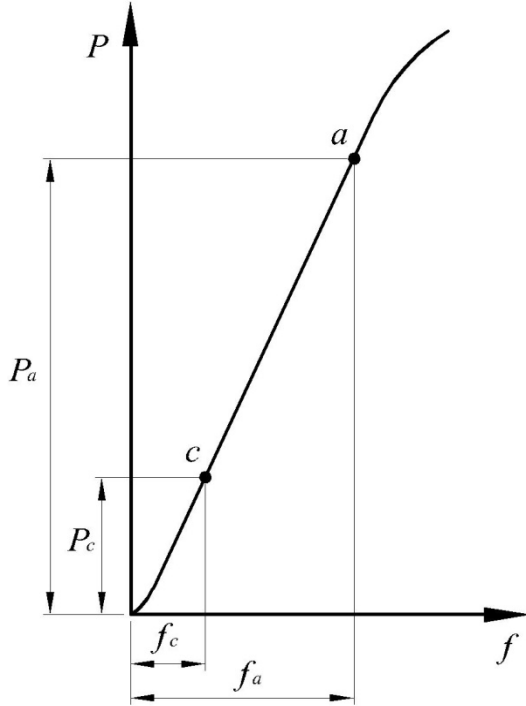


Fig. 2. Functional relation between P and f

For the points a and c within the proportionality limits of the dependence of P from f , we can write the following:

$$P_a = \frac{4Ebh^3}{l^3} \cdot f_a, \quad (6)$$

$$P_c = \frac{4Ebh^3}{l^3} \cdot f_c \quad (7)$$

$$P_a - P_c = \frac{4Ebh^3}{l^3} \cdot (f_a - f_c) \quad (8)$$

From (8) we draw the dependency relation for Young's static modulus – E :

$$E = \frac{(P_a - P_c)l^3}{4bh^3(f_a - f_c)} \quad (9)$$

The dynamic elastic modulus is determined on the basis of the standard ASTM E1876 - 09 [3] by the equation:

$$E_d = 0.9465(mf_f^2 / b)(L^3 / t^3)T_1 \quad (10)$$

where:

E_d - is Young's dynamic modulus;

m - girder's weight;

b - width of the girder;

L - length of the girder;

t - thickness of the girder;

f_f - fundamental resonance frequency at bending;

T_1 - geometric correction coefficient.

The fundamental resonance frequency at bending is measured according to standard. [3]. This is shown on Fig.3

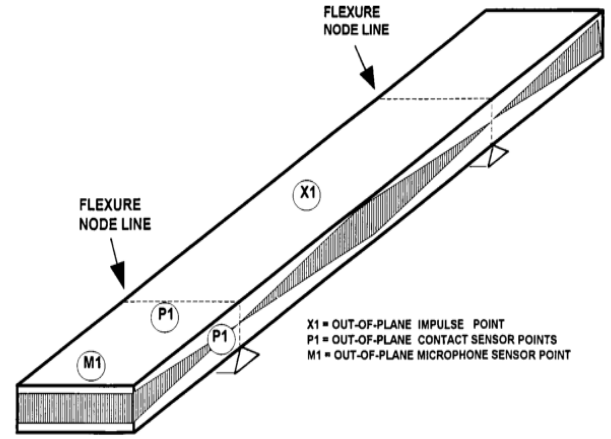


Fig. 3. Setting for measuring the vibrations at bending

The geometric correction coefficient T_1 we determine depending on the ratio L/t , as follows:

$$L/t > 20 \quad - \quad T_1 = [1.000 + 6.585(t/L)^2] \quad (11)$$

$L/t < 20$, then the value of T_1 is calculated by:

$$T_1 = 1.000 + 6.585(1 + 0.0752\mu + 0.8109\mu^2)(t/L)^2 - 0.868(t/L)^4 - \frac{8.340(1 + 0.2023\mu + 2.173\mu^2)(t/L)^4}{1.000 + 6338(1 + 0.1408\mu + 1.536\mu^2)(t/L)^2} \quad (12)$$

If Poisson's ratio μ is unknown, we have to assume its initial hypothetical value μ_0 . Subsequently, it can be determined by an iteration procedure based on calculation of the dynamic shear modulus G_d , according to the algorithm shown on Fig.4.

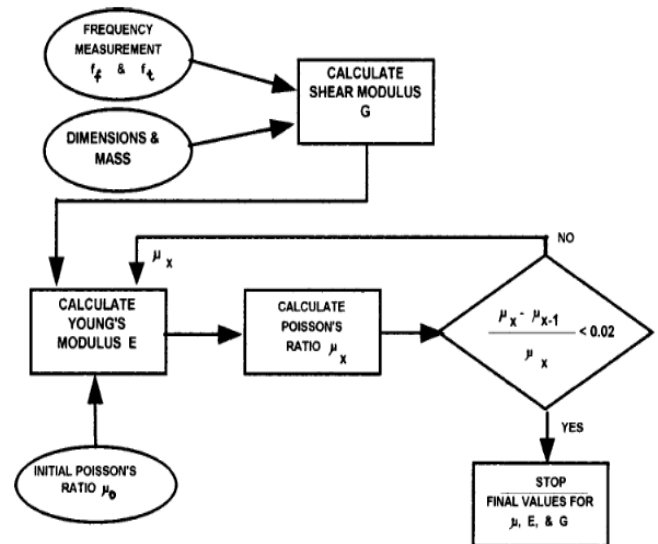


Fig. 4. Algorithm for determining Poisson's ratio

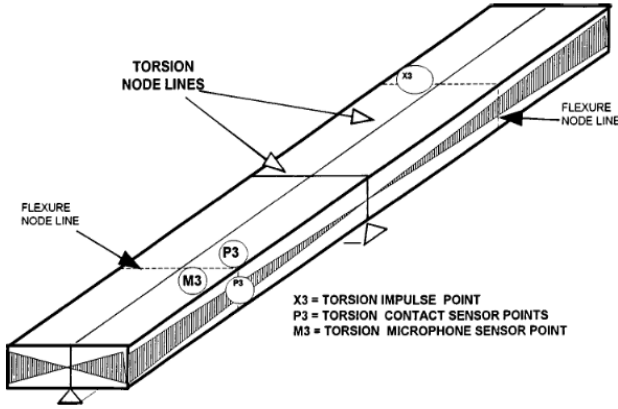


Fig. 5. Measurement of the vibrations of torque

The dynamic shear modulus G_d is calculated by the following equation:

$$G_d = 4(Lmf_T^2 / bt)[B / (1 + A)] \quad (14)$$

where:

f_T - is the fundamental resonance frequency at torque. It is measured according to the standard [3]. This is shown on Fig. 5.

A and B are correction empiric coefficients for width and thickness.

$$B = \frac{b/t + t/b}{t/(t/b) - 2.52(t/b)^2 + 0.21(t/b)^6} \quad (15)$$

$$A = \frac{[0.5062 - 0.8776(b/t) + 0.3504(b/t)^2 - 0.0078(b/t)^3]}{[12.03(b/t) + 9.892(b/t)^2]} \quad (16)$$

In the case of isotropic behavior of the material, Poisson's ratio is calculated, by the relation:

$$\mu = (E_d / 2G_d) - 1 \quad (17)$$



Fig.6. Experimental setting for determining the static modulus

3. Results from the experiments

The experimental samples are with the shape of a rectangular parallelepiped (girder type) with dimensions 30x30x350 mm, in compliance with the common standardized norms. The number of polymer concrete composites is 15, of which a total of 45 bodies are made (3 pieces of each composite).

The stand for determining the static modulus of linear deformation E at bending is shown on Fig. 6. The experimental setting for determining the dynamic modulus E_d is shown on Fig. 7. The frequencies f_f and f_T are determined by the frequency ranges, obtained from impulse excitation of the samples, Fig.8 and 9.

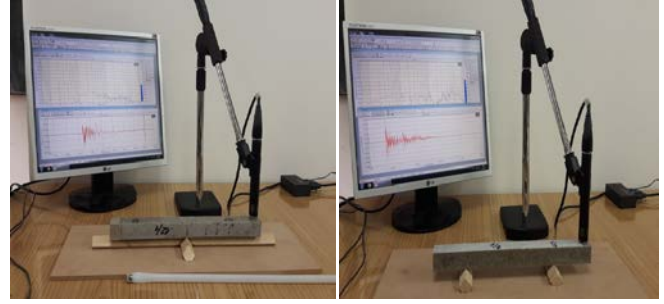


Fig. 7. Experimental setting for determining f_f and f_T



Fig. 8. Frequency range for f_f sample 3.2



Fig. 9. Frequency range for f_T sample 3.2

The obtained experimental results for the average values of the modules of linear deformation for each experimental polymer concrete composite, after calculation, are presented in Table 1.

Average module values **Table 1**

sample №	E [Pa]	E_d [Pa]	relative difference %
1	23,9E+9	33,7E+9	29,05%
2	19,2E+9	28,9E+9	33,58%
3	36,8E+9	42,3E+9	13,09%
4	19,5E+9	29,0E+9	32,93%
5	16,7E+9	25,0E+9	33,26%
6	12,2E+9	19,3E+9	36,50%
7	15,1E+9	23,7E+9	36,25%
8	14,1E+9	22,3E+9	36,86%
9	17,4E+9	24,5E+9	28,84%
10	16,2E+9	26,7E+9	39,20%
11	15,8E+9	24,4E+9	35,26%
12	13,8E+9	22,1E+9	37,60%
13	18,3E+9	27,8E+9	33,96%
14	25,8E+9	34,3E+9	24,72%
15	16,3E+9	24,3E+9	32,88%

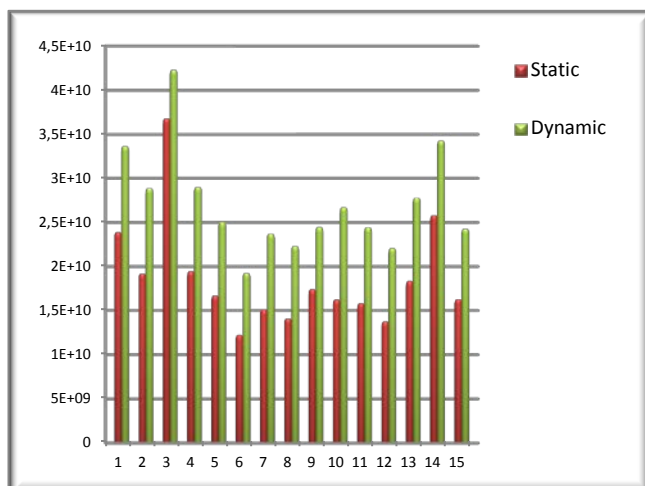


Fig. 9. Histogram of the static and dynamic modules

The results of the articles may be summarized as follows:

The quantitative values of the elasticity modules of 15 different polymer concrete composites are obtained. In determining the static modulus E by the method of three-point bending, the saggings f_i and loadings P_i are measured experimentally.

In determining the dynamic modulus, the fundamental bending f_f and torque f_T frequencies of the tested samples were measured with the help of the experimental modal analysis, within their frequency ranges.

A comparative analysis was carried out of the values of the static and dynamic composite elasticity modules. The composite with maximum values of the modulus was determined.

The opportunities for obtaining credible information about the modules for this type of composites, on the basis of the proposed methodology and measurement equipment, are real and adequate.

4. Conclusions

Analyzing the obtained values of the static and dynamic Young's modules, we reach to the conclusion that the values of the dynamic modules are higher with 30÷40%. This can be explained with the occurring relaxation processes with a wide range of relaxation periods. Even in mechanical oscillations with considerable frequency, relaxation processes occur in polymer concrete composites with relaxation periods that are shorter than the oscillation period. These relaxation processes cause an aftereffect expressed in further deformation that is as smaller, as the greater is the frequency of the mechanical oscillations.

Composite no.3 has maximum values of the modules E and E_d .

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GRANULATED FOAM GLASS. PRODUCTION, PHYSICAL AND MECHANICAL PROPERTIES

ГРАНУЛИРАНО ПЕНОСТЪКЛО. ПРОИЗВОДСТВО. ФИЗИЧНИ И МЕХАНИЧНИ ХАРАКТЕРИСТИКИ

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Abstract: The article is devoted to an up-to-date material for heat and sound insulation. A technology for production of granulated foam glass from glass waste is developed based on horizontal model of machine for foaming, tempering and cooling granules. Mechanical and thermo-physical indicators for obtained foam glass are investigated.

Keywords: GRANULATED FOAM GLASS, FOAMING AGENTS, PROPERTIES

1. Introduction

The foam glass is widely used in the form of blocks, plates, grain of different sizes, components for thermal insulation in construction, shipbuilding, in machines, aggregates as well as for sound insulation, filters and others. [1].

The foam glass exceeds the alternative materials (foam-diatomite, ceramic-perlite, perlite cement bond, vermiculite, concrete, mineral wool, glass wool, polystyrene, polyurethane, ceramic fibers) in thermal conductivity, mechanical strength, moisture absorption. It is applicable in construction, shipbuilding, chemical industry, nuclear industry, military industry and other economic sectors.

2. Production of granulated foam glass

A technology for production of granulated foam glass used for construction of roads, bridges, roof coverings, insulation for foundations of buildings and insulation for pipelines transporting fluids laid in the ground, is in process of development at the IMSETHC-BAS, Sofia.

The subject of the development is creation and testing of technology based on the horizontal model for receiving pieces of foam glass granules (50÷100) mm, [2]. Its implementation requires purposeful activity in the following areas:

- Establishing the optimum batch compositions, binding additives and pressure by rolling to obtain granules of foam glass;
- Formation of granules of different burden compounds;
- Development and testing heat-technical characteristics of the process, forming granules and foaming components;
- The way for evenly feed the granulated mixture on the conveying belt, position 8 (Fig.1);
- Identifying the main characteristics of the horizontal installation: the rate of heating and cooling in the technological areas (speed of belt movement; type and thickness of the coating on the border granulated foam glass and metal surface of the conveyor belt, aiming high performance and quality of the final product);
- Development of design documentation for additional modules to horizontal installation for production of foam glass, [3, 4, 5];
- Development of modules to a horizontal installation;
- Conducting technological experiments for settings of the installation and putting into operation an experimental station for production of granulated foam glasses;
- Investigating the physical and mechanical characteristics of the obtained granulated foam glasses.

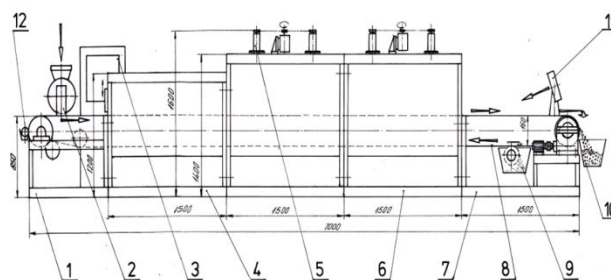


Fig.1 General view of model of horizontal installation for production of granulated foam glass:

1. Adoption station;
2. Submission dosing device for grains;
3. Control panel;
4. Heat and foaming section of the furnace;
5. Vent-hole of temperature station;
6. Tempering sections of the furnace;
7. Rear station;
8. Conveying belt;
9. Clean modular device;
10. Receiving module finished granules;
11. Control panel;
12. Coated modular device.

Fig. 2 shows a preliminary graphics of "the law" for heating the material in the process of obtaining granulated foam glass in horizontal installation.

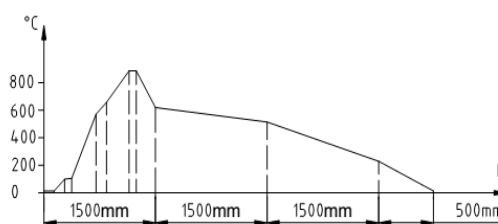


Fig. 2 Law for the heating the material in the furnace

The dosed on the conveyor belt unfoamed granules enter the working space of the blowing module (furnace with operating temperature, which is adjusted to set values depending on the granules composition). The parameters of blowing module (length) and the speed of the belt movement (20÷30 cm/min) allow a certain point on the moving conveyor belt to stay in the furnace for (6÷10) min. The granules with an initial diameter (5÷10 or 10÷14) mm, increase their diameter 2,5 times. The stabilizing module of the furnace is with circulating air which lowers the temperature of foamed granules from 580°C to 300°C, at that lowering the temperature granules stay for (15÷20)min, followed by cooling to a temperature (60÷70)°C at which temperature they enter the receiver.

3. Preparation of granules from burdening compounds A, B and C for the Study of physical-chemical and thermo-physical properties of the finished granulated foam glass [6]

Raw material is processed to fractions of 85 % under 63 μm sieve and 15 % fraction (63÷100) μm . Using part of the glass powder having specific surface of (5500-6000) cm^2/g micro-granules of three compositions were prepared, by passing the mixture of glass powder and blowing agent through a series of sieves:

A/ Mixture containing 100 % powder from household glass waste fraction under 63 μm – 85 % fraction (63÷100) μm - 15% with 1 % glycerin and 3 % water glass with module 1,2÷1,3 over 100 % and granulated with water glass with module 0,6÷0,8;

B/ Mixture containing 100 % powder from household glass waste, fraction under 63 μm - 85% and fraction (63÷100) μm and 5 % CaCO_3 over 100 % granulated with water-glass with module (0,8 ÷ 1,0);

C/ Mixture containing 100 % powder of glass waste - fraction under 63 μm - 85% and fraction (63 ÷ 100) μm , 15% with added 1 % glycerin, 3 % water glass with module (1.2÷1.3) and 11 % Al_2O_3 - fraction below 50 μm , granulated with water glass with a module (0.6 ÷ 0.8).

Granules from compositions **A**, **B** and **C** are manufactured using disk granulator. The granules of each composition are separated by a set of sieves on fractions: (2÷4; 4÷6; 6÷8; 8÷10, 10÷12, 12÷14 and 14÷16) mm. These are the raw materials which physicochemical and thermo-physical properties were tested. Experimentally is determined the time of placing the granulated mixture until the time at which optimum foaming is achieved without a process of overheating and occurrence of unwanted deformations, i.e. spoiling the spherical form. In cyclic entering into the furnace of cold granules from burdening compound, initial temperature of 800°C decreases to (770÷780)°C. At this temperature begins process of foaming. This process continues with increasing temperature up to 800°C. The time for this increase matches with the time for passing of the overall technological process, approximately (5÷8) min, depending on the glass granules size. The tempering is carried out as foam glass granules pass to another zone of the furnace or at (600÷610)°C for (2÷4)min and then – to zone for cooling to room temperature.

4. Preparation of samples for conducting physical-chemical, physical-mechanical and thermos-physical tests

Foam glass granules with volumes, which allowing elaboration of samples with suitable dimensions are prepared from compositions **A**, **B** and **C**.

For testings as compressive strength and flexural strength the sizes of specimens comply with the requirements of BDS EN 60068 (MIL STD-810-G): cubes, cylinders and rectangular prisms, with proper form and defined dimensions. To determine the heat conduction are made plates with dimensions (140 x 140 x 50) mm with parallel and flat surfaces.

For measuring the coefficient of linear thermal expansion are made cylinders with diameter of 7 mm and length 100 mm from the three compositions **A**, **B** and **C**.

5. Tests for determination of compressive strength, bending strength, heat conduction, water absorption, of compositions A, B and C [7]

5.1 Compressive strength - determined using a tensile / pressure testing machine, as the sample is evenly loaded with a force perpendicular to its surface.

The samples are cubes with length edge of 40 mm. The average values of measured compressive strength of compositions **A**, **B** and **C** are given in Table 1.

$$(1) \sigma_H = P / S_0,$$

where: σ_H - compressive strength, [MPa]; **P** - maximum pressure force, [N]; S_0 - cross-section of the test sample, [mm^2].

5.2 Bending strength - since the samples have rectangular section, the bending strength is calculated by the formula:

$$(2) \sigma_{or} = 3PL / 2bh^2,$$

where: σ_{or} - bending strength, [MPa]; **P** – maximum load of the sample, [N]; **L** - the distance between axes of rollers, [mm]; **b** - width of the test specimen, [mm]; **h** - thickness of test specimen, [mm].

For each composition are made 5 samples with length: **L** = 100 mm, width: **b** = 30 mm and height: **h** = 15 mm. The results for compositions **A**, **B** and **C** are presented in Table 1.

5.3 Factor of heat conduction – it is calculated by the formula:

$$(3) \lambda = Q \cdot a / s \cdot \tau (t_1 - t_2), [W/(m.K)],$$

where: **Q** - the quantity of transferred heat, [W]; **a** - distance between the end surfaces of the sample, perpendicular to the direction of the heat flow, [cm]; **s** - cross-section of the test sample [cm^2]; τ - time, [s]; t_1, t_2 - temperatures of the end surfaces, [$^{\circ}\text{C}$], whereupon $t_1 > t_2$.

The data for the measuring factor of heat conduction are given in Table 1.

5.4 Water absorption – it is calculated by the formula:

$$(4) B = (g_1 - g_0) / g_0 \cdot 100, [\%],$$

where: **B** - water absorption, [%]; g_1 – weight of the saturated with water sample, [g]; g_0 - weight of the dry sample, before the saturation of the sample with water, [g].

Saturation of samples (with a volume of about 5 cm^3) is carried out as pieces of foam glass granules were placed in boiling water for 1 hour. During boiling is restored evaporated water. The test data are given in Table 1.

Table 1

Staff	Compress live strength, MPa	Bending strength, MPa	Factor of heat conduction, W/(m.K)	Water absorption, %
A	4.41	1.76	0.09	0 ÷ 4
B	5.39	2.16	0.12	8 ÷ 9
C	4.90	1.96	0.10	2 ÷ 4

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A COMPARISON OF FRICTION CHARACTERISTICS OF VARIOUS SURFACE TREATED HOT WORK TOOL STEEL

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Abstract: Wear, is one of the most important failure type which reduces the service lifetime and increases the maintenance cost of machines. In addition, wear is directly related with surface properties and friction characteristics. So, researchers focus on various surface treatments to reduce the wear and its cost. Plasma nitriding is one of surface treatment which forms a few hundred micron diffusion zone and a surface inter-metallic phases with moderate surface hardness. PVD coating is another method which ensures ultra-hard and a few micron surface layers. In the present study, AISI H13 hot work tool steel substrate was surface treated with various methods, such as plasma nitriding, PVD coating and duplex treatment. Effects of the treatments on the surface roughness, hardness were investigated. Additionally, microstructural analysis was performed to present the effect of treatments on the surface microstructures. Furthermore, friction characteristics were investigated using ball on disk testing machine and wear tracks were discussed using optical microscopy.

Keywords: PLASMA NITRIDING, PVD COATING, DUPLEX TREATMENT, FRICTION

1. Introduction

Wear, is one of the most important factor that effects the service lifetime, occurs due to relative motion of contacting components. Plasma nitriding (PN) and physical vapour deposition (PVD) are mostly used to enhance the wear resistant of steels. While the PN forms different zones (white zone, nitriding zone) which are very thick compared to PVD, the PVD forms only a few microns thick layer, which is harder than PN, on the surface of steel. PVD coating on a relatively soft base material causes an inconsistency related with hardness values. Therefore, to form a transition zone makes the service time longer. These techniques have some advantages/disadvantages on each other. So, application of these techniques together is a way to eliminate the disadvantages of each [1–5]

In this study, PN and PVD applied on AISI H13 steel with different combinations. Friction characteristics were obtained via ball on disk test. Effect of the surface treatments on surface hardness also measured. Additionally, microstructural examinations and microhardness measurement were performed

2. Material and Method

The chemical composition of AISI H13 steel used as base material can be seen in Table 1. Prior to all surface treatments as-received steel was heat treated using the schedule given in Fig. 1. PN parameters were given in Table 2. Two different PVD coatings were selected and application was made by Oerlikon Balzers and Ionbond Tinkap. Information about PVD coating were presented in Table 3. Nomenclature of the samples produced using different treatments were given in Table 4.

Table 1. Chemical composition of hot work tool steel used in this study

DIN	AISI	C	Si	Mn	Cr	Mo	V
1.2344	H13	0,39	1,00	0,40	5,30	1,40	0,90

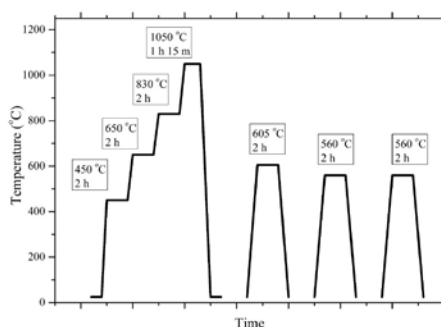


Fig. 1. Tempering heat treatment schedule

Table 2. PN parameters

Temperature	Time	Gas Ratios	Pressure
500 °C	10 h	H ₂ /N ₂ (¼)	250 Pa

Table 3. PVD coatings

	Coating thickness(µm)	Friction coefficient	Micro hardness (HV)	Coating color
Ionbond (CrN)	1-4	0,40	2400-2600	Silver
Oerlikon Balzers (TiAlN)	depend on the application	0,30-0,35	3300	Purple-Gray

Table 4. Nomenclature of samples

Specimen	Treatment
S00	As received
S10	Tempered
S11	Tempered + PVD (CrN)
S12	Tempered + PVD (TiAlN)
S20	Tempered + PN
S21	Tempered + PN + PVD (CrN)
S22	Tempered + PN + PVD (TiAlN)

To reveal the effect of PN on surface roughness, all samples were polished after tempering to obtain a certain surface roughness. Surface polishing was also carried out before PVD coating to enhance the bonding capability of PVD layer. Samples were analyzed optically for microstructural changes and possible defects using a Nikon DIC microscope with a Clemex image. Vickers microhardness measurements were performed on a DUROLINE-M microhardness tester. Ball on disk testing machine was used to compare friction characteristics of different treatments. Friction speed and load were 4.45 mm/s and 5.82 N, respectively. Maximum and mean Hertz contact pressure was calculated as 1.76 GPa and 1.16 GPa, respectively.

3. Result and Discussion

Surface roughness of different heat treated samples were measured and given in Fig. 2. While PN and CrN PVD coating on tempered sample decreased the surface roughness slightly, PVD TiAlN coating increased. Similar to this findings, the difference of the surface roughness of CrN and TiAlN coating was reported by Björk et al.[6]. Although Batista et al. [7] reported that duplex coating occurred higher surface roughness, in the present study, no important difference was observed. It can be related with polishing

procedure after PN. In addition, duplex treatment increased the surface hardness in both case.

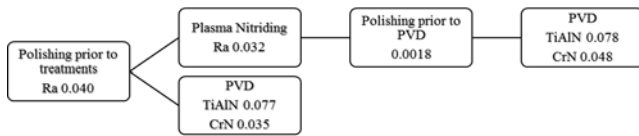


Figure 2. Surface roughness of samples

All obtained microstructures can be seen in Fig 3. As tempered sample contained martensitic microstructure with carbides of elements such as Cr, Mo and V. Plasma nitrided samples shows different zones including white zone and nitrided transition zone. Diffusion zone measured as 100 μm roughly. CrN PVD coating formed 1-2 μm thick non-uniform layer, while TiAlN PVD coating formed 3-5 μm thick uniform layer. It should also be noted that TiAlN PVD coating had an interlayer to enhance the adherence on coating layer on substrate.

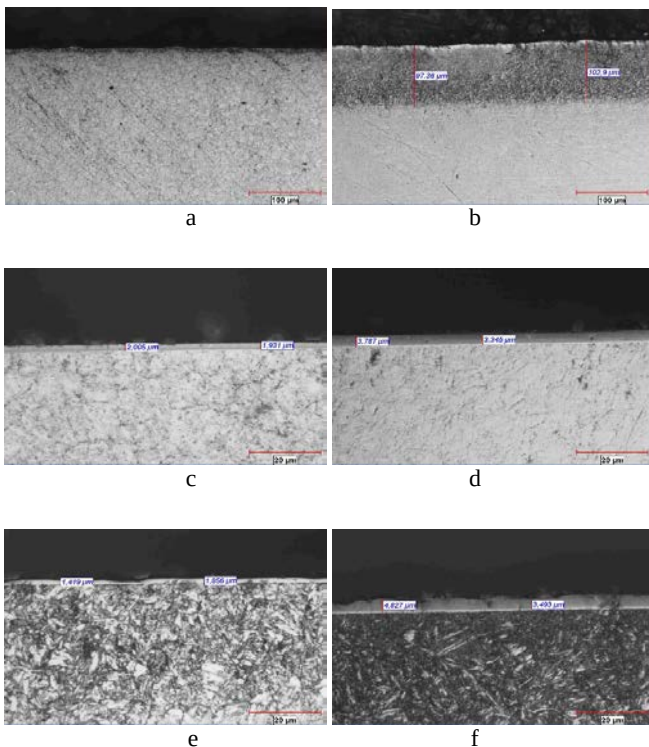


Figure 3. Obtained microstructures of samples a)S10 b)S20 c)S11 d)S12 e)S21 f)S22

The surface hardness values of the investigated samples are presented in Fig. 4. It can be seen that the untreated sample had a hardness of 227 $\text{HV}_{0.025}$. The surface hardness of the tempered sample reached 616 $\text{HV}_{0.025}$ because of the formation of tempered martensite. The micro hardness profile of plasma nitrided sample shown in Fig.5. The combination of tempering and plasma nitriding processes increased the surface hardness of the untreated material to 1059 $\text{HV}_{0.025}$, as a result of the formation of the compound layer containing extremely hard iron nitrides. According to DIN 50 190/3, nitriding depth defined as the depth which shows 50 HV higher hardness than base metal. The nitriding depth calculated as 160 μm . The surface hardness of the TiAlN coating on the plasma nitrided layer reached 3320 $\text{HV}_{0.025}$, which was approximately around 15 times that of the untreated substrate and 3 times that of the plasma nitrided layer. Similarly, the surface hardness of the CrN coating on the plasma nitrided layer showed a significant increase (2424 $\text{HV}_{0.025}$), specifically, approximately 11 times that of the untreated substrate and 2 times that of the plasma nitrided layer.

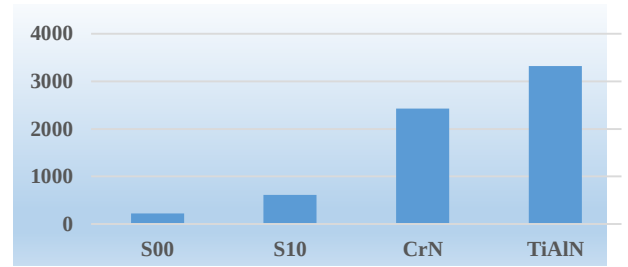


Figure 4. Microhardness values of samples

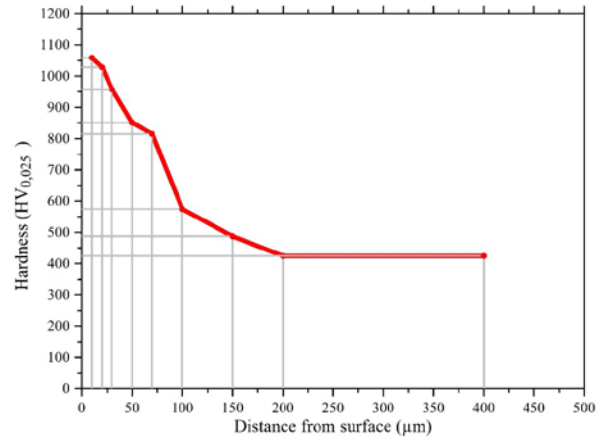


Figure 5. Microhardness profile of plasma nitrided sample

Friction coefficient change versus distance can be seen in Fig. 6. Friction coefficient values start from a specific value related with surface conditions created by heat treatment. After a while, due to activated wear mechanisms (abrasive and fatigue wear in this study) friction coefficients increased and reached a specific value. PVD coated samples exhibited better performance than plasma nitrided sample, while duplex treated samples showed the best performance. TiAlN PVD coated samples exhibited superior behavior over CrN PVD coated samples. Taktak et al. [8] reported that PN decreased the friction coefficient. Additionally, as can be seen from the change of friction coefficient graphs, applied load did not crack the coating layers when the load placed on the sample. Wear track was formed by wear mechanisms.

Optical micrographs of wear tracks were presented in Fig. 6. The width of the wear tracks are in accordance with friction characteristics. The mostly worn sample, as can be easily predicted, was the tempered one. While, plasma nitrided sample shows excessive wear, PVD coated and duplex treated samples show almost no wear.

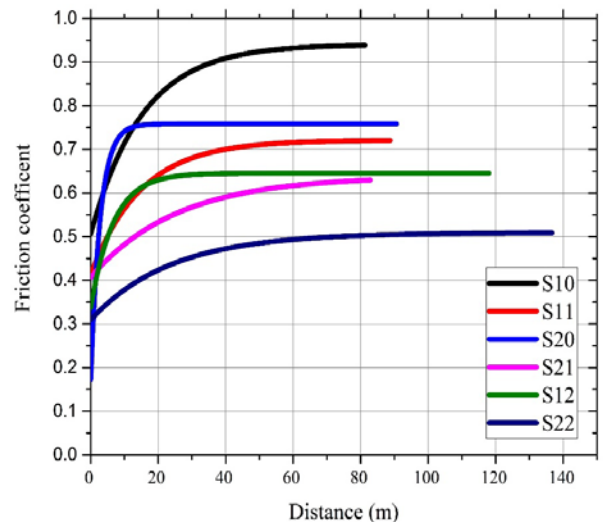


Figure 5. Friction coefficients of tested samples

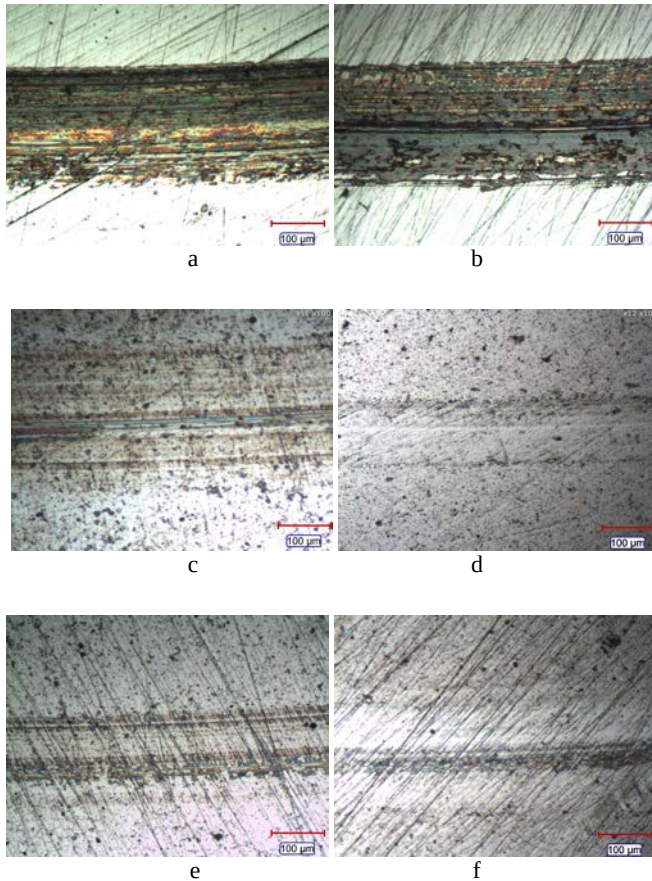


Figure 7. Optical micrographs of wear track of samples a)S10 b)S20 c)S11 d)S12 e)S21 f)S22

4. Conclusion

The present study was focused on the friction characteristics of AISI H13 tool steels treated with duplex treatments. From this investigation, the following conclusions can be derived.

- The use of a plasma nitriding process on the tempered H13 steel increased its surface hardness to approximately 4.7 times that of the untreated substrates and approximately 1.8 times that of the tempered samples.
- The use of a plasma nitriding process prior to PVD coating played an important role in the increase of the surface hardness. Surface hardness of the TiAlN-coated sample was approximately 15 times higher than that of the untreated substrates and approximately 3 times than that of the plasma nitrided layers. However, the surface hardness of the CrN-coated sample was approximately 11 times higher than that of the untreated substrates and approximately 2 times greater than that of the plasma nitrided layers.
- PN enhanced friction properties of tempered steel while PVD coated samples exhibited better performance than plasma nitrided samples.
- TiAlN PVD duplex treatment exhibited the best performance among all treatments.

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NEW IRON OXIDES/HYDROXIDES BIOMATERIALS FOR APPLICATION IN ELECTRONICS AND MEDICINE

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НОВИ БИОМАТЕРИАЛИ ОТ ЖЕЛЕЗНИ ОКСИДИ/ХИДРОКСИДИ ЗА ПРИЛОЖЕНИЕ В ЕЛЕКТРОНИКАТА И МЕДИЦИНАТА

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Abstract: Biotechnologies could supply inexpensive, environment friendly and effective materials for new nonstandard and concurrent solutions. The present work's focus was on acquiring new physical data for application in electronics and medicine of biogenic materials obtained due to the metabolism in laboratory conditions of iron-oxidizing bacteria from the genus *Leptothrix*. Powders and coatings on glass samples were under investigation. XRD and Raman Spectroscopy analysis of the data collected show that the Fe^{2+} -depending from the growth media could transformed into Fe^{3+} or $\text{Fe}^{2.5+}$ in the form of two types of oxides/hydroxides such as magnetite (Fe_3O_4) and lepidocrocite ($\gamma\text{-FeOOH}$), all with nanostructured morphology. Biotechnology for obtaining one biogenic iron phosphate hydroxide (ferrian giniite) - $(\text{Fe}_5(\text{PO}_4)_4(\text{OH})_3 \cdot 2\text{H}_2\text{O})$ was also developed. The ferrian magnetic material is dispersed in the walls of biogenic tubular structures. The average particle's size and crystalline structures of bio-products were investigated. Results based on PPMS measurements on the magnetic properties were reported. The new biogenic materials showed superparamagnetic behaviour and high sensitivity to electromagnetic radiation and have real potential for application in electronic and information technologies.

Keywords: IRON OXIDES/HYDROXIDES BIOMATERIALS, IRON-OXIDIZING BACTERIA, LEPTOTHRIX, BIO-MAGNETIT, BIO-LEPIDOCROCITE, BIO-FERRIAN GINIITE, SUPERPARAMAGNETIC BIO-PARTICALS

1. Introduction

Molecular electronics, biosensors and biocomputers are scientific areas which demonstrate extremely fast progress in the last decade. Nanosized biogenic iron oxides(oxy)hydroxides are promising alternative to polymers applied in molecular electronics and reveal new perspectives. Biotechnologies could supply inexpensive, environment friendly and effective materials for new nonstandard and concurrent solutions. Superparamagnetic iron oxide hybrid nanoparticles having high bio-compatibility have been used "in vivo" experiments in NMR, for better contrast, repair of tissue. The first patent for artificially preparation of nanosized biogenic tubes [1] appeared in 2012. A large part of Earth's sedimentary iron deposits can be attributed directly or indirectly to microbial activity [2]. Microorganisms, so called Fe-oxidizing bacteria (FeOB) are able to oxidize Fe^{2+} and form Fe^{3+} bioproducts. Ones of the bacteria competing with the kinetically more favorable abiotic Fe^{2+} oxidation are those from the group *Sphaerotilus* – *Leptothrix*. As a result of the bacteria-mediated iron oxidation, red/orange microbial mats are formed, which comprise iron (oxy)hydroxides in a polysaccharide matrix in the specific form of tubular sheaths, with the exact composition of the sheaths yet to be fully determined. The question about the function of the iron oxidation by the bacteria is still open for debate due to the lack of sufficient information and difficulties with their cultivation under laboratory conditions.

The present work's focus was on acquiring new physical data for application in electronics and medicine of the biogenic material obtained due to the metabolism in laboratory conditions of FeOB from the genus *Leptothrix*. Depending on the media for cultivation used, we obtained different iron-containing nanostructured materials with crystalline identification as lepidocrocite, magnetite and ferrian giniite. Powders, coatings and bio-micro tubes from these materials were under investigation.

2. Materials and Methods

The procedure for sample preparation under laboratory conditions is outlined in Fig 1.

In this research we used a pure culture of the bacteria from the genus *Leptothrix* isolated from a natural stream in Vitosha Mountain, located at altitude of 1783 m [3, 4]. Two types of growth media for obtaining desired biogenic oxides and tubular structures (sheaths) were used: Adler's medium (AM) [5] and Silicon Iron Glucose Peptone medium (SIGP) [6] and Fernbach, Roux and Erlenmeyer flasks for the cultivation under static conditions. The cultivation is described in detail by Angelova R. et al. [3, 4]. The specific shape of the selected vessels provides a high volume and surface for aeration of the culture fluid in order to allow the bacteria to grow. The flasks were inoculated (10% v/v) and cultivated at a temperature of 20 °C. The additional iron source (iron cuttings) was added in each growth medium. The iron source for AM and SIGP media was ammonium iron (II) sulfate $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ and ferrous sulfate $(\text{FeSO}_4 \cdot 7\text{H}_2\text{O})$, respectively.

Biotechnology for obtaining of a biogenic iron phosphate hydroxide (ferrian giniite) - $(\text{Fe}_5(\text{PO}_4)_4(\text{OH})_3 \cdot 2\text{H}_2\text{O})$ was developed based on the laboratory cultivated *Leptothrix* bacteria in SIGP with $\text{Pb}(\text{NO}_3)_2$. The technology is growing up to the patent documents and will be called provisory SIGPP.

To characterize the biogenic materials obtained we used X-ray diffraction, Raman spectroscopy and TEM imaging. The magnetic measurements were performed on a Physical Properties Measurement System (PPMS) up to 1 T. All samples were obtained after 40 days of cultivation, filtering and drying of the biomass at room temperature, excluding the method for obtaining ferrian giniite, which the cultivation period was 14 days.

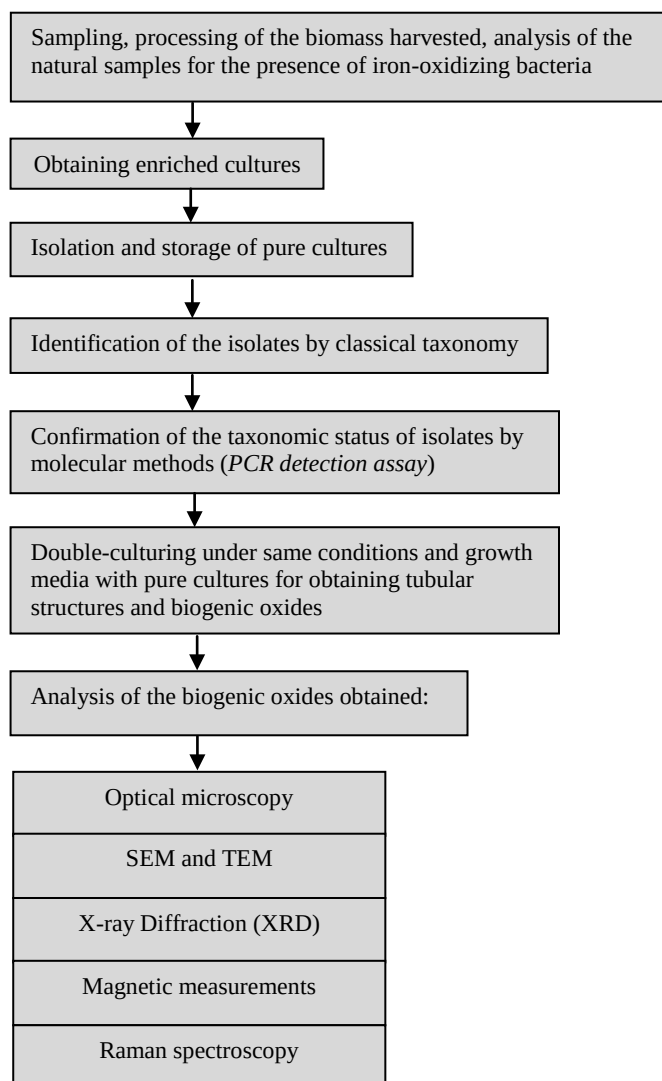


Fig. 1 Schematic illustration of the preparation and characterization procedure of the investigated materials [14]

3. Results and Discussion

Choosing modified Adler's medium was a decision based on assessing the results from the quantity of biomass obtained in grams, in connection with the growth time in days, with these media showing the best results. In order to achieve any type of application, the quantities of iron-bearing material obtained biotically have to compete with those obtained inorganically. Figure 2a) shows SEM images of FeOB cultivated in AM. The typical morphology of cells can be seen with shape characteristic of the genus *Leptothrix*. Hollow sheaths were revealed by SEM and light microscopy only in SIGP medium. However, none of the samples investigated contained the typical *Leptothrix* sp. hollow sheaths, as revealed by numerous SEM and light microscopy observations, even under longer periods of cultivation. This confirmed some previous investigations, which concluded that these bacteria, in most cases, lose their ability to form iron-containing sheaths when they have been cultivated in vitro [6, 5]. The cultivation in AM did not lead to the formation of such structures, but we were able to prepare sufficient quantities of biomass with ochre coloring obviously containing various types of iron oxides/(oxy) hydroxides.

The cultivation in SIGP medium gives the possibilities due to the metabolism of *Leptothrix* sp. to obtain submicron and nanosized sheaths. The cultivation in SIGPP medium allows to form tubular structures with nanosized spheres of a biogenic iron phosphate hydroxide (ferrian giniite) - $(\text{Fe}_5(\text{PO}_4)_4(\text{OH})_3 \cdot 2\text{H}_2\text{O})$ discreetly

dispersed in the sheath walls. The typical sheaths structures are illustrated in Fig. 2b).

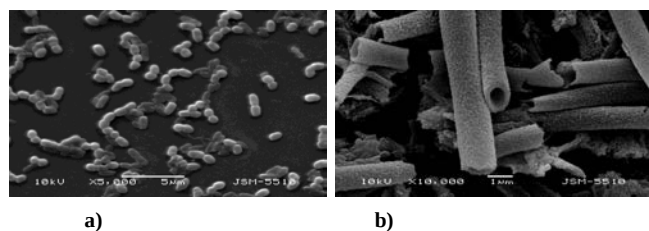


Fig. 2 SEM of bacteria from the genus *Leptothrix* cultivated in a) Adler's medium: panoramic view and b) tubular structures obtained after cultivation in SIGP medium.

The biogenically derived materials from the cultivation were characterized by XRD. Figure 3a) presents XRD spectra of samples obtained from AM. The analysis of the spectra reveals that the phase composition is mixed. For the material collected from the AM-cultivated *Leptothrix* strain, it was as follows: lepidocrocite ($\gamma\text{-FeOOH}$) – 60 %; magnetite (Fe_3O_4) – 22 % and goethite ($\alpha\text{-FeOOH}$) – 18 %. The average size of all particles was calculated independently, all of them being on the nanometer scale. It was as follows: lepidocrocite particles – 30 nm; magnetite particles – 24 nm and goethite particles – 12 nm.

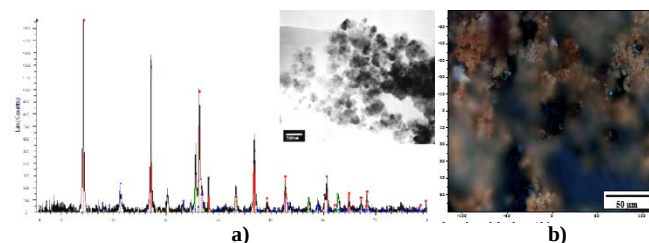


Fig. 3a) XRD spectra of biogenic material obtained in AM after cultivation of *Leptothrix* bacteria and inserted a TEM image of the powder obtained and **b)** Optical microscope photographs of the bacteria-derived material deposited on glass slides from AM.

Glass slides from AM (see Fig. 3b) with different biofilms depending on the growth media used were prepared from FeOB cultivated for 40 days by three drop-wise depositions of collected and filtered biomass. Each layer was dried at room temperature. Thus prepared, the samples were examined by Raman microspectroscopy using a LabRAM HR Visible single spectrometer equipped with a microscope and a Peltier-cooled CCD detector. The 633-nm He-Ne laser line was used for excitation. The Raman measurements were performed at room temperature.

Raman spectra of the material are systematized in Table 1 depending in AM growth medium used, the corresponding iron by-products detected and their positions in the Raman signal. We have to note that a strong luminescence signal was also present during the measurements arising from all samples containing iron oxides/(oxy) hydroxides, which greatly impeded our investigation. It stretched over the whole spectral range (100 to 3400 cm^{-1}) of the spectra obtained.

Table 1: Raman scattering data of the biogenic material from AM.

Growth medium (sample)	Raman peak positions detected in sample* (cm^{-1})	Assigned iron oxides/hydroxides**
Adler's medium (AM)	221, 254 , 312, 350, 381, 465 , 530, 650	lepidocrocite ($\gamma\text{-FeO}(\text{OH})$)
	310 , 540, 670	magnetite (Fe_3O_4)

* The prominent peaks positions in the Raman signals detected are shown in bold.

** The assignment of spectra is on the basis of literature data [9,10,11]. Since, to the best of our knowledge, Raman studies on biogenic material have not been performed before, the comparison was made with data for natural and synthetic minerals, corrosion products etc.

The single broad band at 670 cm^{-1} is well-known feature of magnetite (Fe_3O_4). Magnetite's other two very weak bands at 310 and 530 cm^{-1} . Magnetite is a ferrimagnetic material with iron in a mixed valence state. Its structural formula can be written as $(\text{Fe}^{3+})_A[\text{Fe}^{2+}\text{Fe}^{3+}]_B\text{O}_4$, where "A" and "B" are magnetite's tetrahedral and octahedral sublattices. Its appearance in the AM sample to the amount of around 22 % (as calculated from the XRD spectra) of the entire iron-bearing mass cannot straightforward be explained yet. One possible scheme for its formation can be deduced from a small very weak spectral feature, which is not, by any means, characteristic for lepidocrocite. The band at around 460 cm^{-1} is a fingerprint for only one iron-bearing compound, namely, ferrous hydroxide $\text{Fe}(\text{OH})_2$. It cannot be ruled out that these small traces of $\text{Fe}(\text{OH})_2$ are of biogenic origin, because the mechanism of the reactions involving the hydrogen peroxide (H_2O_2) derived from the *Leptothrix* sp. metabolism and the nutrition present are not fully understood, especially on the interface bacterial wall/iron source. Only some tips elevated higher of the biomass level are colored differently from reddish-brown and also in the fact that the 460 cm^{-1} peak is present in the lepidocrocite spectra only. Such small areas with concentrated $\text{Fe}(\text{OH})_2$ can act as centers for nucleation of magnetite. It is more thermodynamically stable than the hydroxide and it evolves most probably via the scheme described by Herane M. et al.[11]. In the scheme summarized there, the formation of magnetite passes through an intermediate phase – green rust, which is unstable in air. No Raman peak positions of goethite were detected, although the XRD spectra of the sample show that it was present, with nearly 18 % of the volume. In our opinion, this is due to the specifics of the Raman spectroscopy probing having to do with the laser beam being focused on spots with diameter below $50\text{ }\mu\text{m}$ in a search for a flat surface. On the other hand the XRD data showing the good crystallinity of the lepidocrocite detected and that the goethite is in poor crystallinity state and really could speak for the trace of him in mix with the lepidocrocite. Material obtained from the AM exhibited a strong response to a weak DC magnetic field, indicative of the magnetite-phase leading role regardless of its lower content. The magnetic properties were investigated by PPMS. For the AM sample, we measured magnetic hysteresis up to 2 T at 4 and 300 K; they are presented in Fig. 4 a) and b). The coercive fields H_c are 73 and 14 Oe at 4 and 300 K, respectively. Magnetization values more typical for magnetite, rather than any antiferromagnetic or paramagnetic material, can be seen. The result shows superparamagnetic behavior of the AM sample at room temperature (300 K - see the inserted Fig.4b). The main contributor to the magnetic behavior of the biogenic product is the nanosized magnetite. The existence of a "none-zero" low coercive - Fig. 4b) field of 14 Oe could be due to the presence of a small amount of magnetite particles with a diameter larger than the superparamagnetic limit (30 nm) for this kind of particles or small contribution from the antiferromagnetic particles. The remanent magnetization (MR) is higher for the lower temperature.

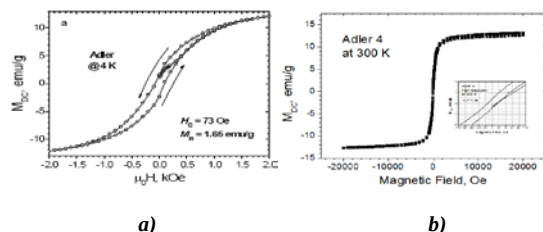


Fig.4 Hysteresis loops $M(H)$ of AM sample at **a)** 4 K and **b)** 300 K.

The sediment from AM sample was dried at 200°C up to 2 hours. Figure 5a) shows the thermal analysis of the AM sample heated up to 1000°C at air with velocity $10^\circ\text{C}/\text{min}$. Strong endothermic effect was observed in the interval $200\text{--}370^\circ\text{C}$ which has summary character including: (i) dehydration, (ii) chemical transformation of $\gamma\text{-FeOOH} \rightarrow \gamma\text{-Fe}_2\text{O}_3$ in the interval $180\text{--}300^\circ\text{C}$ and (iii) burn out of the organic mass - the high level of the weight losses in the sample. Obviously after annealing could awaiting two ferrimagnetic phase in the powder, respectively magnetite Fe_3O_4

(bulk $M_s=93\text{ emu/g}$) and maghemite $\gamma\text{-Fe}_2\text{O}_3$ (bulk $M_s=73\text{--}74\text{ emu/g}$). Magnetic measurements show that the ferrimagnetic phase increases with 71% (inserted M_s in Fig. 5b). The magnetic investigations show that the powder keeps their superparamagnetic behavior. The superparamagnetic nature of the AM biogenic product makes it a perfect candidate for a magnetic component in biocompatible ferrofluids [12], as the currently used Gd-based contrast agents are also paramagnetic in nature.

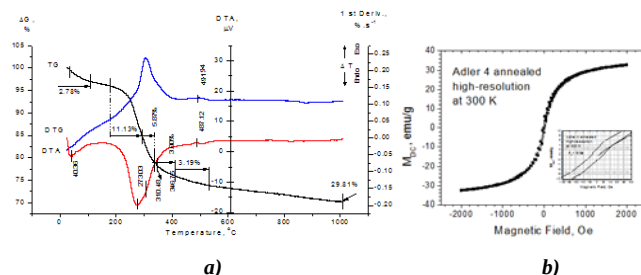


Fig. 5a) DTA, TG and DTG investigation of AM samples and **b)** Hysteresis loop of annealed AM powder.

In the SIGP medium as a result of the bacterial metabolism submicron and nanosized sheaths have been formed (Fig. 6). The XRD pattern (Fig. 6) of the tubes obtained from the modified SIGPP medium shows a single-phase crystalline structure of the inorganic inclusions of a biogenic iron phosphate hydroxide (ferrian-giniite) - $(\text{Fe}_5(\text{PO}_4)_4(\text{OH})_3 \cdot 2\text{H}_2\text{O})$. The ferrian magnetic material is discreetly dispersed in the walls of these biogenic tubular structures. In the scientific literature the magnetic data of this bio-material are very limited.

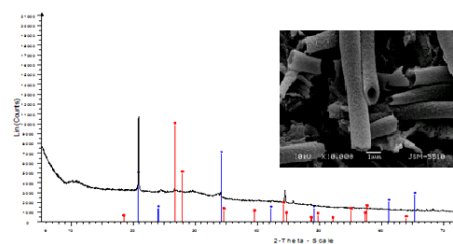


Fig. 6 XRD spectra of biogenic material from SIGPP sample after cultivation of *Leptothrix* bacteria and an inserted image of the typical tubular structures obtained after cultivation in SIGP medium.

Magnetic measurements (Fig. 7a) show a typical ferrimagnetic behavior of the sample with tubular structure. In contrast to the crystals of ferrian-giniite obtained by convenient chemical technologies [13] this bio-product has a weak hysteresis behavior which is probably defined by the nanosized dimension of the particles.

Figure 7b) presents the fitted data of the magnetic saturation with an applied magnetic field $H=1\text{ T}$ and inserted the hysteresis behavior at 1T where the curve goes down up to inverse point. Investigation by PPMS from 4 K up to 80 K show the superparamagnetic hysteresis loops typical for a single domain magnetic particles. After 80 K the magnetic moment abruptly rise but without saturation till the maximum of applied magnetic field.

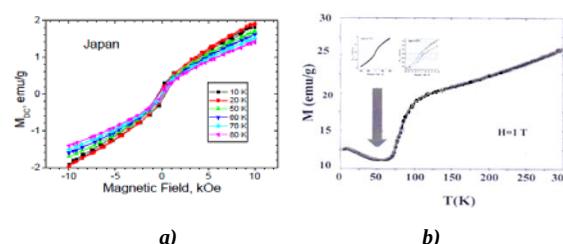


Fig. 7 Magnetic properties of SIGP sample: **a)** Superparamagnetic $M(H)$ up to 80 K and **b)** Non-saturated magnetic moment up to 10 T.

4. Conclusions

Bionanotechnology was employed to produce biogenic nanosized iron oxide powders and nanotubes containing magnetic nanoparticles due to the cultivation of the bacteria from the genus *Leptothrix* under laboratory conditions. The transformation of Fe^{2+} to Fe^{3+} due to bacterial metabolism in the powders was around 78 %, with the rest being found to be in a mixed 2,5 valance state in magnetite. The average size of all particles was calculated independently and found to be on the nanometric scale. The magnetic measurements results showed a superparamagnetic behavior of the iron oxide/hydroxide powders at room temperature (300 K). The main contributor to the magnetic behavior of the biogenic product is the nanosized magnetite and after annealing to 200°C the ferrimagnetic mix from Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$. In the SIGPP sample as a result of the bacteria metabolism submicron and nanosized tubes has been formed with the inorganic inclusions in the walls of a biogenic iron phosphate hydroxide (ferrian giniite) - $(\text{Fe}_5(\text{PO}_4)_4(\text{OH})_3 \cdot 2\text{H}_2\text{O})$. The understanding of the structural and magnetic properties of these materials is important for fundamental research, but also could suggest some development of potential applications in magnetoelectronics and information technologies.

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EFFECT OF METAL PHASE COMPOSITION ON MECHANICAL AND TRIBOLOGICAL PROPERTIES OF Fe-GLASS COMPOSITES

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The effect of metal phase composition in the Fe-based + (2 % glass) powdered composites on the basic mechanical and tribological properties of the composites, made by means of sintering and hot forging, have been investigated. As a basis for the metallic phase of the composite the mixtures of iron powders with additives of graphite, B₄C, BN and Cu at different ratio were used. It was shown that at sintering of metal-glass material the reaction of glass phase with oxides on the surface of iron powder particles takes place, resulting in a change of glass phase chemical composition. The results of materials mechanical properties investigations had shown that the highest strength properties and hardness have the composites with the content of the initial powder mixture of 5% Cu and 2% B₄C, while the best tribological properties have the composites with 2% B₄C, 5% Cu and 1% BN.

Keywords: Fe-GLASS COMPOSITE, SINTERING, HOT FORGING, FRICTION, POWDER, STRENGTH, HARDNESS, TRIBOLOGICAL PROPERTIES.

Introduction

One of the major advantages of powder metallurgy is a capability for manufacturing of pseudoalloys, whose synthesis by conventional casting techniques is almost impossible.

A prime example of such materials are the metal-glass composites, that consist of relatively plastic metal matrix in which the glass inclusions are uniformly distributed [1]. These composites showed high efficiency as the tribological materials when used for the manufacturing for the parts of friction units that operate in diverse and complex mediums (for instance - without lubrication, in vacuum, in abrasive-carrying medium, etc.). Having relatively high hardness and wear resistance, the glass when combined with ductile metal, is an effective component of tribological composites.

Manufacturing of this kind of materials is impossible by casting owing to the large difference in melting points of metal and glass, the coagulation and segregation of the latter by density during the melting process. At the same time, in case of powder metallurgy techniques, in the process of sintering of metal-glass material the glass phase interacts with iron oxides on the surface of the powder particles, that enhances the adhesive bond strength between the components of the mixture. Furthermore, a change in the chemical composition of the glass phase takes place with separation therein of solid chemical compounds that increase the tribological properties of the material due to formation of substantially heterogeneous composite structure.

In most publications devoted to investigations of structure and properties of metal-glass materials, as starting materials to obtain composites with glass the mixtures of unalloyed iron and

carbon powders were used. Besides, as our previous studies had shown [1], the optimum content of the glass phase in the glass-metal composite that provides relatively high durability at a stable value of the friction coefficient and relatively high strength characteristics, is about 5÷7 % (wt.). At the same time, obviously, we can expect an increase in the basic physical and mechanical properties and performance of materials by modifying of metal matrix phase with appropriate additives that enhance both tribological characteristics when using the composite as the friction material.

Thereby, the aim of this article was to investigate the effect of some additives in the composition of the initial powder mixture and technological schemes of metal-glass composites manufacturing on features of structure, basic mechanical and tribological characteristics of the latter.

Materials and methods of the experiment

In the investigations of interaction of iron with glass phase during sintering powders used glass of two kinds were used - pane and "Pirex". Their chemical composition is presented in the table. 1. Glass particle size was 75÷80 microns.

As the metallic phase of the powder mixture iron powder with a particle size of 80-160 microns of industrial purity was used. The glass content in the mixture was 15 % (wt.).

The initial powder mixtures were pressed on the hydraulic press at different compression pressures in order to obtain different initial porosity of the preforms - from 10 to 30 %. Sintering of the preforms was realized in the medium of hydrogen at temperatures of 700, 800, 900, 1000 and 1100 °C.

Table 1

Chemical composition of the used glasses

Kind of the glass	Склад скляної фази, %							t _{subsidence} , °C
	SiO ₂	Na ₂ O	CaO	MgO	Al ₂ O ₃	B ₂ O ₃	K ₂ O	
Pane	71,5	15	8,5	3,5	1,5	--	--	530
"Pirex"	80	4	--	--	2	12	1	620

For investigations of mechanical and tribological characteristics of metal-glass composites initial mixture for synthesis of metal-glass composites were prepared from a mixture of iron powder and 5 % (wt.) pane powder with a particle size of 40 microns, obtained by grinding the sheet glass in a rotary mill. As anti-score additives powders of graphite and boron nitride were used. To increase the hardness and the friction parameters of materials boron carbide was added in a powder mixture, and for improving the strength and thermal conductivity of the base material copper powder was used.

- sintering at 1100 °C, then hot forging on screw press from a temperature of 1100 °C (in argon).

Options of the studied compositions of raw powder mixtures, which are used in the experiment, are presented in the table 2.

The powder mixture obtained by mixing of the powders, the preforms were compacted at a pressure of 700 MPa which were processed by two different technological modes:

- presintering in hydrogen at 800 °C for 1 hour, then second compaction at a pressure of 700-800 MPa and subsequent sintering at 1100 °C;

Table 2

Mixture compositions for manufacturing of metal-glass composites (% wt.)

Elements	Mixture, No.							
	30	31	32	33	34	35	36	37
Glass	5	5	5	5	5	5	5	5
C	2	--	2	--	--	2	2*	--
Cu	--	--	--	--	5	5	--	5
B ₄ C	--	2	--	2	2	--	--	2
BN	--	--	1	1	1	--	--	--
Fe	93	93	92	92	87	88	93	88

* Carbon was used in granules with size of 1-1,5 mm.

Tribological properties of the materials were determined in terms of the butt end friction without lubrication at sliding speeds of 4, 8 and 12 m/s and a load of 1.5 and 2.1 MPa. As a counter body 0,65 % C – 1 % Mn steel, hardened to 52 ÷ 55 HRC was used. During the test, the coefficient of friction and wear rate were estimated.

Experimental results and their discussion

Results of the study of glass particles saturation with base metal during sintering had shown that glass particles changes the color, that denotes on their active saturation with metal ions.

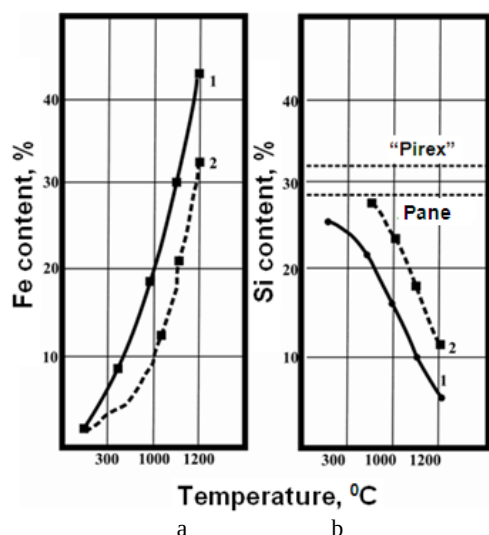


Fig. 1. Effect of sintering temperature on the average content of iron (a) and silicon (b) in glass phase of sintered composite: 1 – pane; 2 – «Pirex» glasses

Increase of sintering temperature assists the growth of the intensity of base metal ions dissolution in the glass. As a consequence of changes in glass chemical composition of sintered Fe-glass materials the increase in the average microhardness of glass particles takes place. With increasing of sintering temperature the microhardness values of glass particles increases too (fig. 2).

Examination of sintered samples microstructure has shown the effect of formation of crystalline phase in glass particles (fig. 3), that usually starts from the surface of a particle. The columnar crystals, formed in the surface layer, with increasing of sintering temperature and exposure time grow deep into the glass.

Analysis of results for evaluation of strength and hardness of sintered and hot forged metal-glass materials (fig. 4) had showed that almost for all of the investigated composites values of these characteristics increase when used boron carbide in material composition (samples No. 31, 33, 34 and 37) as a result of boron carbide dissociation when heated to temperatures that exceed 1050 ÷ 1100 °C [2]. The use of copper in the initial powder mixture (samples 34, 35 and 37) increases the hardness and strength of the materials too due to formation of solid solution after sintering. Infusion in the mixture of boron nitride (samples

The results for investigation of iron and silicon content in glass particles depending on the sintering temperature is shown on fig. 1.

As can be seen from the figure, with increasing of sintering temperature saturation of glass with metal increases. Significant effect on the saturation of glass with Fe makes the kind of the glass and its melting temperature. Thus, in the glass-metal samples with "Pireks" glass its saturation with metal and reduction of silicon content in the glass is less intensive than in the pane glass. This is because the "Pireks" glass has a higher viscosity at the same temperatures than pane glass, hereupon the contact between glass and metal deteriorates and mobility of melt glass reduces.

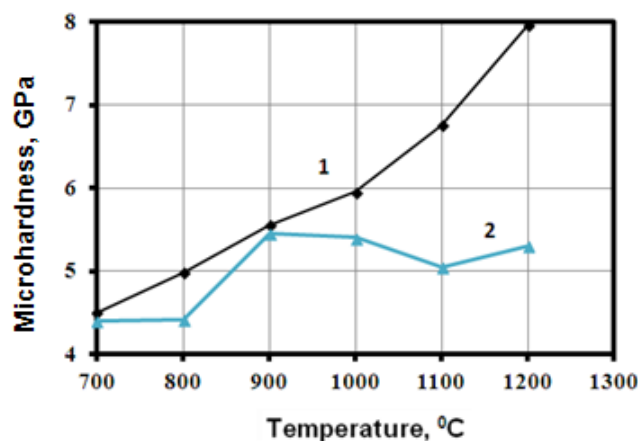


Fig. 2. Effect of sintering temperature on microhardness of glass phase for Fe-glass composites with pane (1) and "Pirex" (2) glasses

32, 33 and 34) leads to the desired effect of reduction of the composites strength and hardness because the latter is virtually insoluble in the iron-carbon alloy during sintering, but presenting itself the antiwelding solid lubricant leads to a significant decrease of level of adhesion force between the friction material and the material of the counter body that supports the smooth friction and reducing wear of the friction surfaces.

The using of hot forging had led to expected significant increase in both strength and hardness of the samples compared to the same materials composition produced by sintering without forging.

The results of investigations of the sintered composites tribological characteristics had showed that the best wear properties had the materials which included boron carbide in the initial powder mixture (No. 31, 33, 34 and 37) due to synthesis of solid boride phases in the sintering process [2]. Among them the best wear properties had the composites alloyed with copper (No. 33, 34 and 37) (fig. 5,a). The single-valued influence of boron nitride according to wear characteristics was not revealed.

For all the compositions of alloys wear rate increase with increasing of sliding speed from 4 to 12 m/s was

established. Increasing of contact friction pressure from 1.5 to 2.1 MPa also leads to a certain increase in the degree of wear level (fig. 5,b).

Analysis of the evaluation of friction coefficients (fig. 5,c) showed a significantly lower dependence of this parameter on the composition of the initial powder mixture, but much more sensitive compared to the characteristic of wear proof properties

dependent on sliding speed. As can be seen from the presented on fig. 5,c data, if the sliding speeds of 4-8 m/s friction coefficient is in the range of 0,35-0,44 (higher values correspond to lower the sliding velocity), then with increasing of slip velocity to 12 m/s values of the friction coefficient is reduced to 0.28-0.36. Increasing contact pressure to 2.1 MPa very little effect on the behavior of the friction coefficient (fig. 5,d).

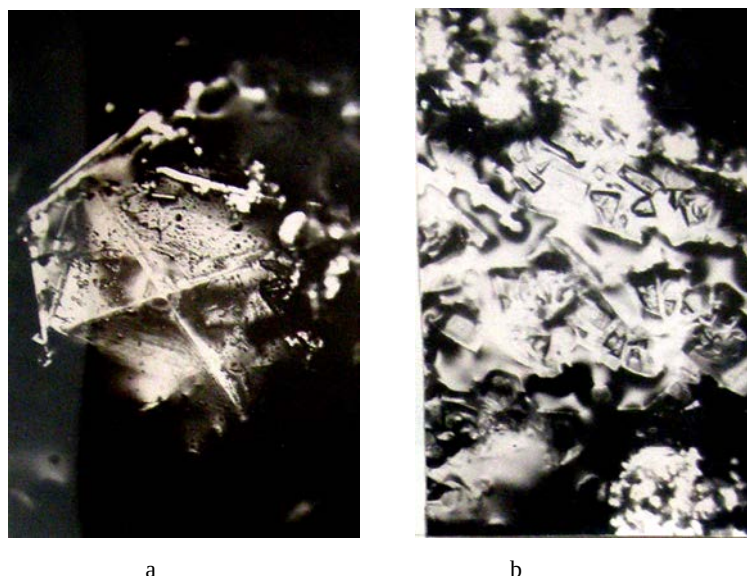


Fig 3. Types of glass crystals, synthesized at sintering of metall-glass composites with pine (a) and "Pirex" (b) glasses

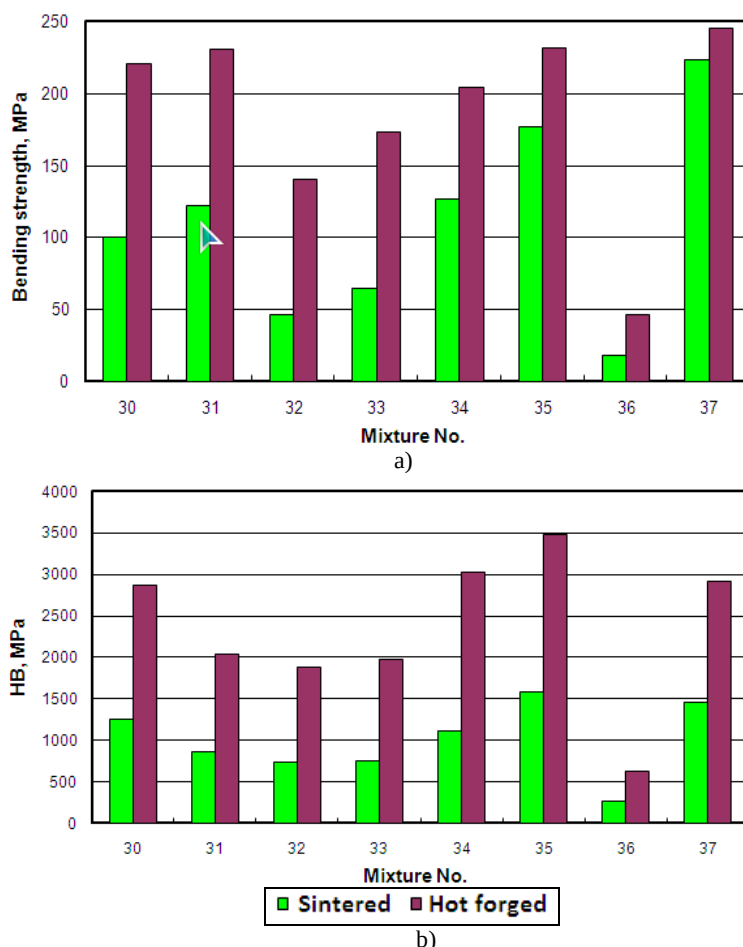
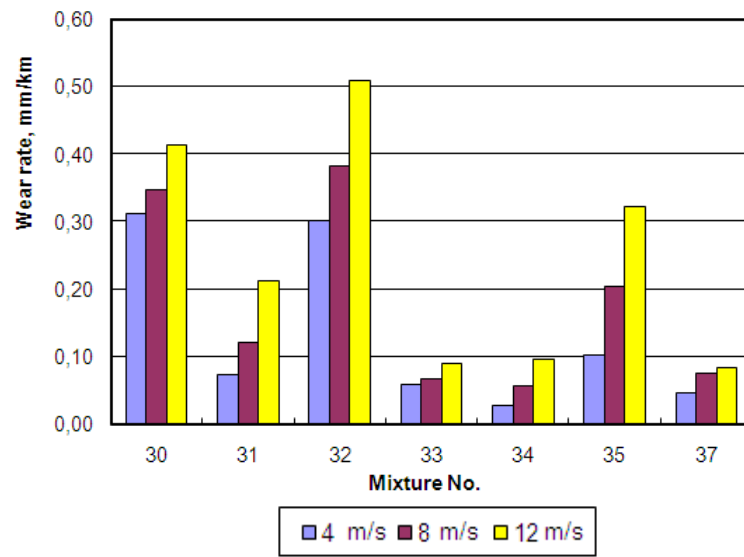
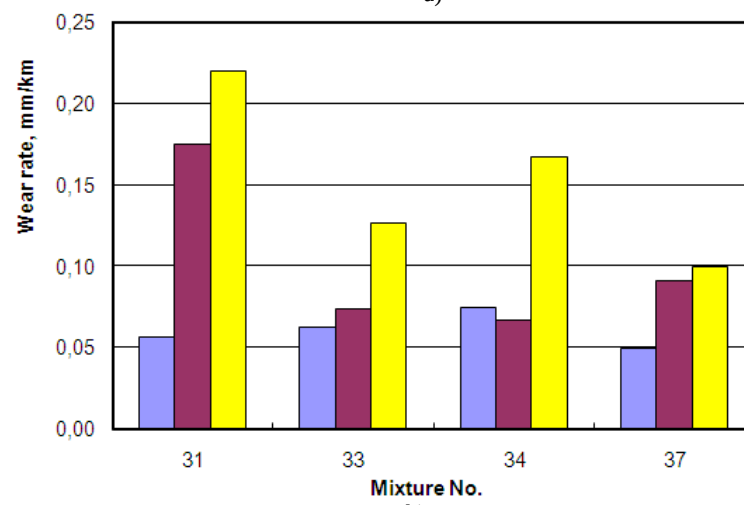


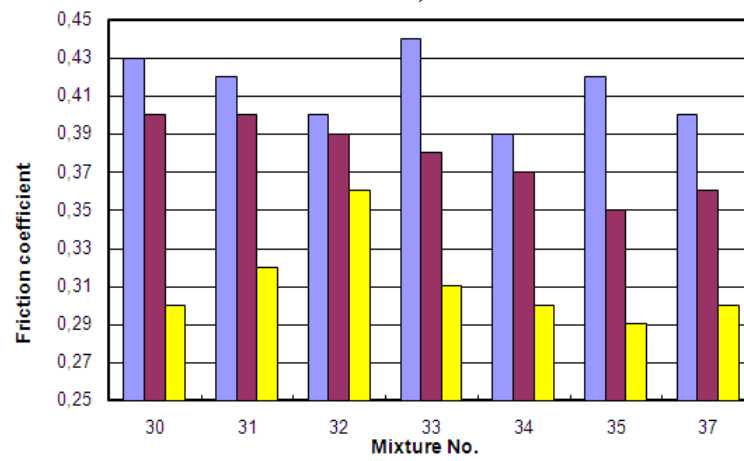
Fig. 4. Bending strength (a) and Brinelle hardness (b) of sintered and hot forged materials, manufactured from powder mixtures of different compositions (see table 1)



a)



b)



c)

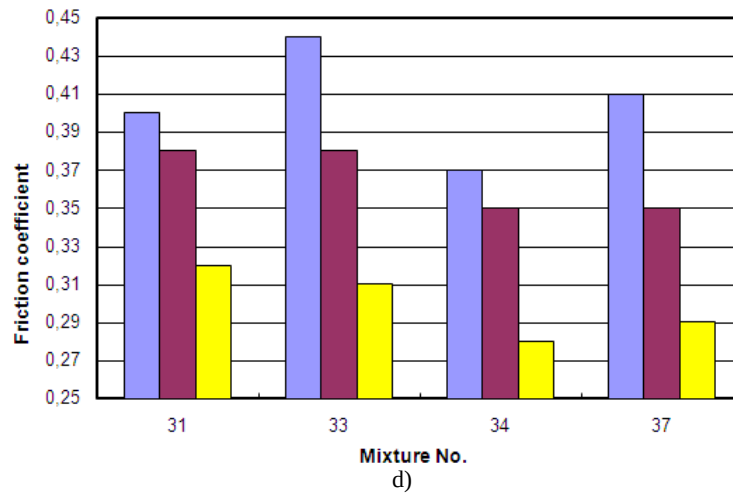
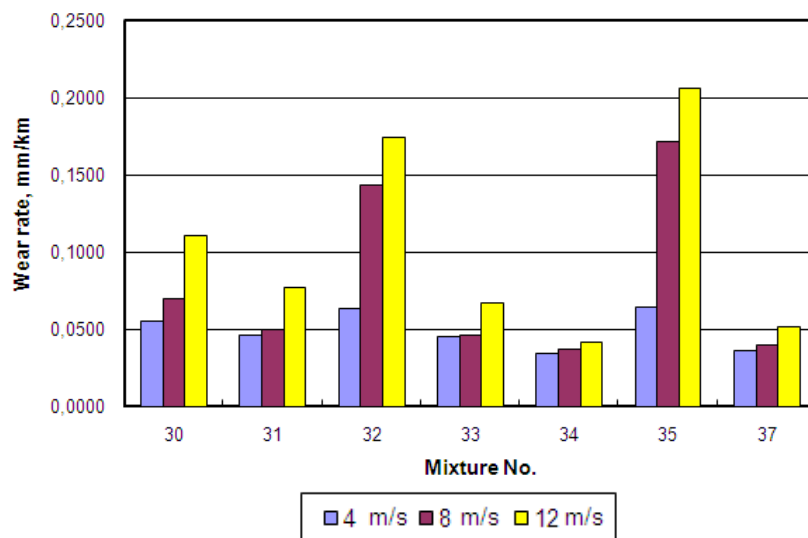


Fig. 5. Tribological properties of sintered and repressed materials manufactured from powder mixtures of different compositions; contact pressure: a, c - 1,5 MPa; b, d - 2,1 MPa

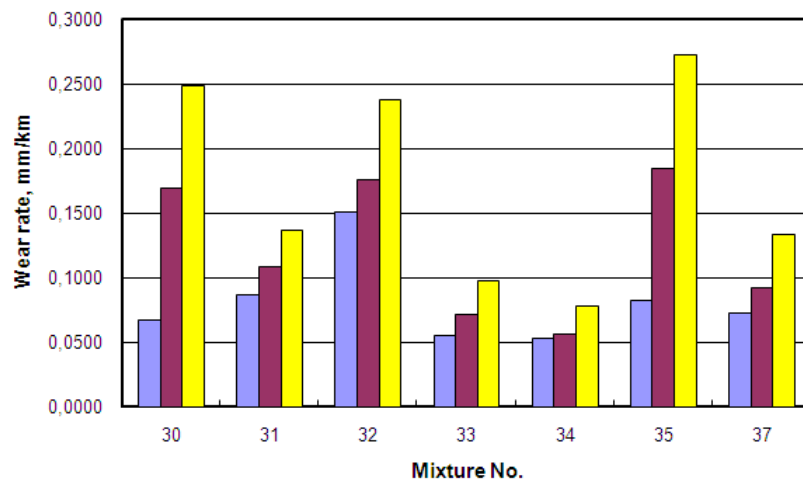
In case of using of hot forging for production of the metal-glass composites a similar regularities with respect to the effect of the initial powder mixture composition on the wear resistance of the material (fig. 6,a) was observed. At the same time, comparison of the absolute values of wear rate indicates that hot forging allows to reduce substantially (for some compositions by more than half) the wear rate of materials in comparison with

analogous compositions obtained by sintering and cold repressing.

Unlike the wear resistance, the values of friction coefficient of hot forged composites small differ from these properties of sintered and repressed material of similar composition.



a)



b)

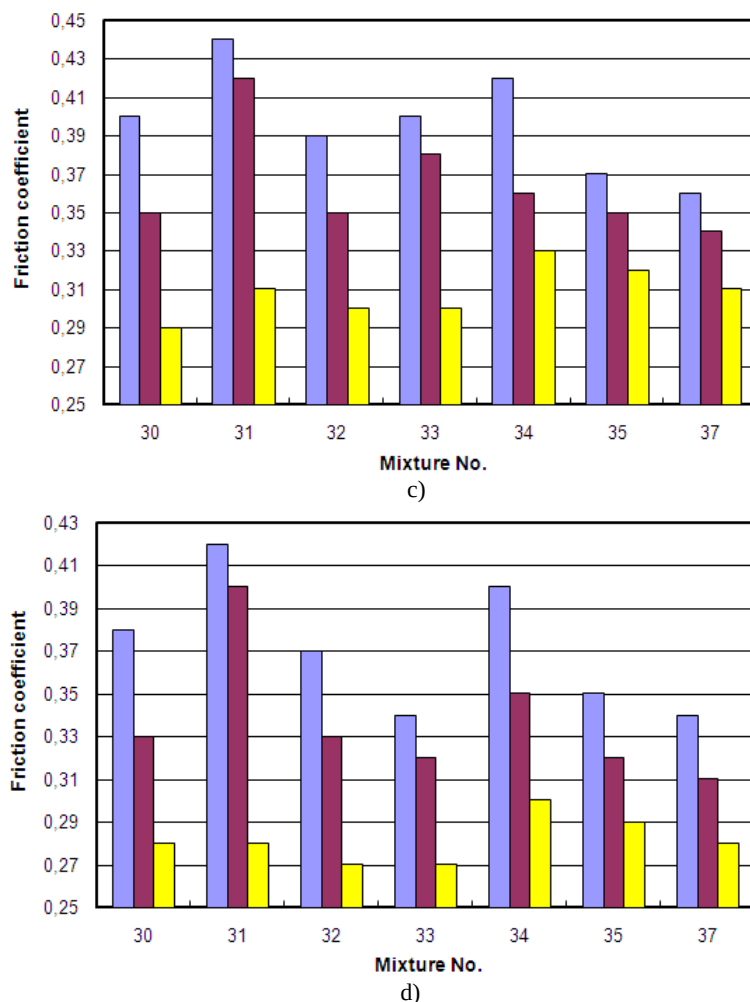


Fig. 6. Tribological properties of hot forged materials manufactured from powder mixtures of different compositions; contact pressure: a, c - 1,5 MPa; b, d - 2,1 MPa

Conclusions

- 1) The highest values for the mechanical properties of the sintered and hot forged composites have the materials made of powder mixtures containing 5% glass, 5% copper and 2% of carbide former components of mixture (graphite, boron carbide).
- 2) The results of investigations of the tribological properties of metal-glass composites leads to the conclusion about the perspectives of their use as a friction material for use in a wide range of sliding speeds.
- 3) Modification of the metal matrix phase with boron carbide provides significant improvement of wear resistance of the material and stable coefficient of friction in the range of 0,27-0,42.
- 4) The use of hot forging promotes significant increase in wear resistance of the material compared with sintered and repressed but little effect on variation of friction coefficient.

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COMPARATIVE STUDY OF TRIBOCORROSION PROPERTIES OF SOME BIO-BASED MATERIALS IN SIMULATED ARTIFICIAL SALIVA

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Abstract:

In this study, the tribocorrosion behavior of the materials utilized in manufacturing of biomedical implants (Ti-6Al-4V and Co-Cr alloys) was studied in a laboratory simulated artificial saliva (SAS) solution by using a linear reciprocating ball-on-plate tribometer with an integrated electrochemical cell. The open circuit potential (OCP) and the friction coefficient were monitored during the reciprocating ball-on-plate test. During the OCP tribocorrosion tests, the generated wear debris was transferred into the SAS and increased its optical density of the solution along with large scatters in the OCP potential and friction coefficient. In accordance with its poor tribocorrosion performance, Ti-6Al-4V alloy provided a large amount of wear debris transfer into the SAS along with the heavy metal attachment to the contact surface of the alumina ball and heavy fluctuations in the OCP potential and friction coefficient values during the sliding. This suggests that the Co-Cr alloy has a higher load bearing capacity than the Ti-6Al-4V alloy.

Keywords: BIO-BASED MATERIALS, Co-Cr, Ti-6Al-4V, TRIBOCORROSION TEST.

1. Introduction

Titanium (Ti) and cobalt chrome (Co-Cr) metal alloys are the most attractive metallic materials for biomedical applications (dental implants and orthopedic prostheses) essentially because these alloys generally present good biocompatibility, good mechanical properties, good corrosion resistance combined with low inflammatory potential [1]. However, poor tribocorrosion behavior of metallic biomaterials in the body fluids has continued to be a major concern [2]. Besides, the presence of particulate corrosion and wear products in the tissues adjacent to the implant can cause various osteoblast integrity issues, which may lead to reduction of osseointegration [3 - 5]. Hence, selection of a metal implant needing both excellent resistance to corrosion, and mechanical properties that satisfy the tribological stresses placed on it during function in a fluid system is indispensable.

In the present investigation, I have conducted a comparative study on the tribocorrosion behavior of Ti-6Al-4V and Co-Cr alloys for biomedical applications, and thereby suggests an appropriate material for biomedical implants.

2. Methods and Procedures

The tribocorrosion behavior of the Ti-6Al-4V and Co-Cr alloys was studied in simulated artificial saliva (SAS) solution by using a ball-on-disk reciprocating tribometer coupled with a three electrode electrochemical cell, which is illustrated in Fig. 1. The Ti-6Al-4V and Co-Cr alloys were served as the working electrode (WE), the saturated calomel electrode (SCE) was the reference electrode (RE) and the platinum electrode was the counter electrode (CE). Reciprocating sliding wear tests were performed in a reciprocating mode with a 1.7 cm s^{-1} sliding rate under 10 N applied load for 45 min. The counter body was an alumina ball with 10 mm diameter. The choice of alumina ball as the counter body was made because of its high hardness, high wear resistance, chemical inertness and electrical insulating properties. The ball holder was made of a polymeric material to prevent the corrosion effects. During the test, alloy with an area of 2 cm^2 was exposed to the corrosive electrolyte. The wear scars were also evaluated by a Scanning Electron Microscopy (SEM). Finally, contact surfaces of alumina balls were examined using an Optical Microscope (OM). After the OCP tribocorrosion tests, microhardness measurements were performed in the wear scar and on the unworn surface of alloys by using the Shimadzu HMV microhardness tester with a Vickers pyramid indenter under indentation load of 100 g.

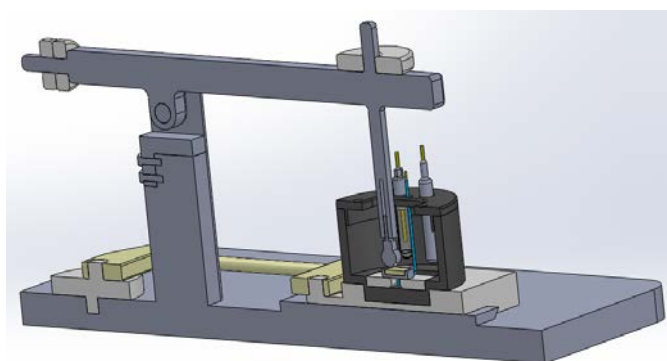


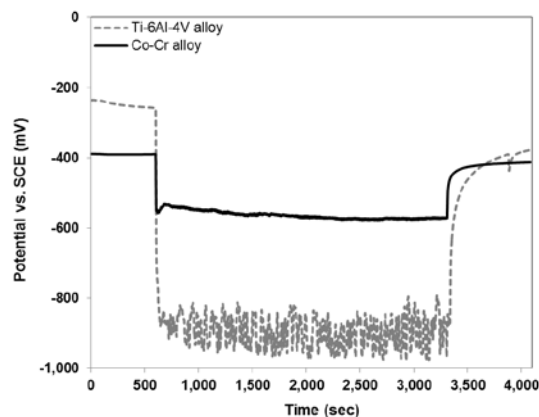
Fig. 1 Schematic representation of reciprocating tribometer coupled with electrochemical cell.

By the end of the OCP tribocorrosion tests, optical densities of SAS solution were measured using a UV-Spectrophotometer. Freshly prepared SAS was analysed as the reference to evaluate the optical densities of solutions. After the spectrophotometric measurement of freshly prepared SAS solution by using fix visible light (wave length: 250 nm), the data obtained was automatically adjusted to zero in order to determine the optical densities of the SAS solutions utilized in the OCP tribocorrosion tests [4].

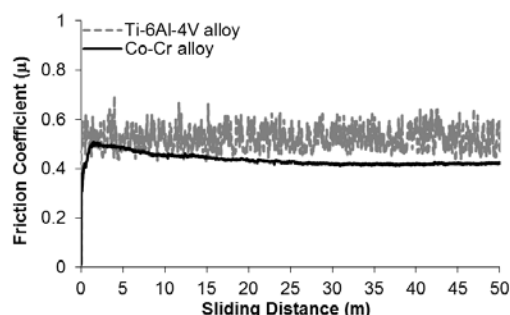
3. Results and Discussions

The evolution of the open circuit potential (OCP) with time before, during, and after the sliding are given in Fig. 2 for Ti-6Al-4V and Co-Cr alloys, together with the friction coefficient values obtained during the sliding. When rubbing is started, the OCP shifts to more cathodic values (-858 mV vs SCE in Ti-6Al-4V alloy, -550 mV vs SCE in Co-Cr alloy) when compared to the initial potential (-257 mV vs SCE in Ti-6Al-4V alloy, -391 mV vs SCE in Co-Cr alloy). When the evolution of OCP is considered (Fig. 2 a), it can be seen that the Co-Cr alloy presented a smaller drop on the potential on the onset of the sliding as compared to the Ti-6Al-4V alloy. During the sliding, the OCP of Co-Cr alloy maintains at around -550 mV, which nobler than Ti-6Al-4V alloy (in between -850 mV and -950 mV). Thus, the OCP drop and fluctuation reflect the extent of depassivation caused by the counter ball [5]. Once rubbing stops, depassivation in the wear scar ceases and the OCP recovers the initial value established before rubbing. Similar behavior has also been reported in the literature [6]. On the other hand, Fig. 2 b shows the evolution of the friction coefficient with sliding distance for the Ti-6Al-4V and Co-Cr alloys under the lubrication of saliva solution. As shown in Fig. 2 b, the Ti-6Al-4V alloy showed friction coefficient values in the range of 0.4 – 0.6 with relatively large

fluctuations; however the friction coefficient of the Co-Cr alloy reached a steady state value of about 0.42 with minor fluctuations and its friction coefficient was slightly lower than the Ti-6Al-4V alloy. The considerable scatters in the OCP potential and friction coefficient values were evident for Ti-6Al-4V alloy through out the testing period (Fig. 2), which can be attributed to the abrasive effect of wear products removed from the worn surface [5, 6].



(a)



(b)

Fig. 2 The evolution of the (a) OCP and (b) friction coefficients monitored as a function of sliding distance during the OCP tribocorrosion tests.

Fig. 3 presents representative SEM images of the worn surfaces of the Ti-6Al-4V and Co-Cr alloys at the end of the OCP tribocorrosion tests. It is clearly seen that the width of the wear scar of Co-Cr alloy was narrower than that of the Ti-6Al-4V alloy. The wear scar of Ti-6Al-4V alloy showed a combination of abrasive and adhesive wear mechanism as evidenced by the scratches with a very large plastic flow over the entire scar length and microcrack perpendicular to the sliding direction (see the arrow in Fig. 3 a). This large plastic flow resulted in work hardening as confirmed by the microhardness values listed in Table 1. According to the difference in microhardness values obtained inside and outside of the wear scar after the OCP tribocorrosion test (Table 1), the extent of hardening for the Ti-6Al-4V alloy was higher than the Co-Cr alloy. This hardening mechanism may also contribute to the formation of the microcrack on the worn surface of Ti-6Al-4V alloy. Similar behaviour was reported by Doni et al. [6] for Ti-6Al-4V/alumina ball tribo-pair immersed in an 8 g/l of NaCl solution. In comparison, the worn surface topography of the Co-Cr alloy was characterized by a mild abrasive wear mechanism (Fig. 3 b). Therefore, it can be said that the differences on the wear mechanisms of both alloys could be attributed to the differences in hardness and repassivation kinetics inside the wear scar.

During the tribocorrosion test for the Ti-6Al-4V alloy, noticeable amounts of wear debris in the SAS solution were detected even with a naked eye. As illustrated in Fig. 4, the wear debris generated from Co-Cr alloy was very few as compared to the Ti-6Al-4V alloy in accordance with its better tribocorrosion performance.

Table 1: Microhardness values measured after tribocorrosion tests at OCP.

Alloys	Microhardness (HV _{0.1})	
	Outside ^a	Inside ^b
Ti-6Al-4V	355±14	416±6
Co-Cr	516±14	525±8

^aOutside – Measurements made outside of the wear track.

^bInside – Measurements made inside of the wear track.

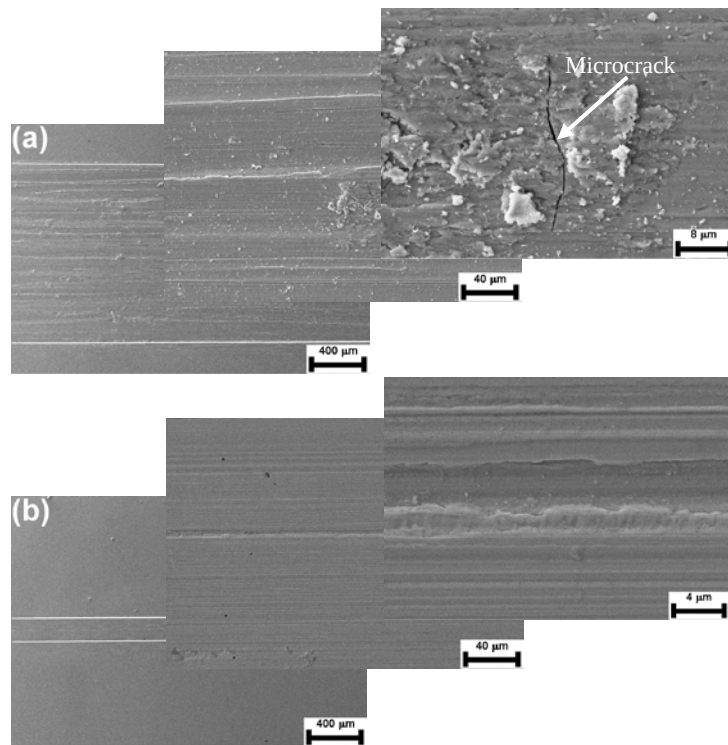


Fig. 3 SEM pictures of the wear tracks after the OCP tribocorrosion tests of the (a) Ti-6Al-4V and (b) Co-Cr alloys (low and high magnifications).

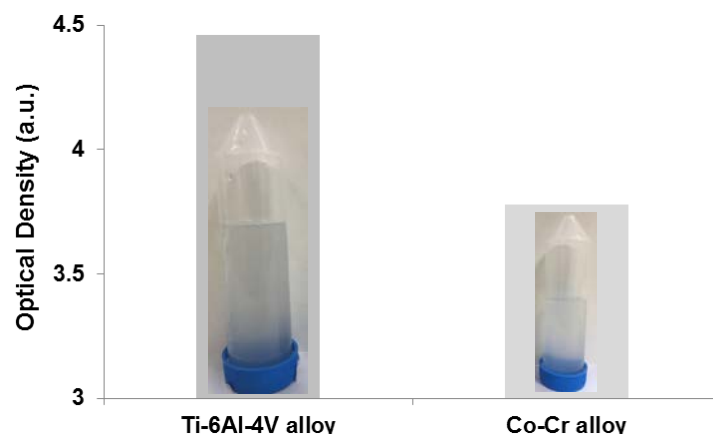


Fig. 4 The variation of optical density of SAS solution utilized in the OCP tribocorrosion tests.

The alumina balls were investigated using OM to identify possible damage (Fig. 5). Wear of Ti-6Al-4V alloy caused darkening at the contact surface of the alumina counter ball (Fig. 5 a). This behavior could be related to the accumulation of wear debris in the SAS solution. On the other hand, some material transfer from the Co-Cr alloy was observed on the alumina ball (Fig. 5 b).

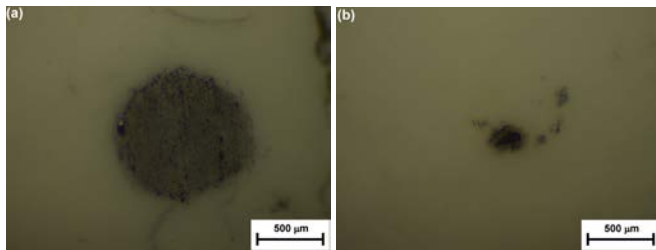


Fig. 5 OM images of the alumina balls after sliding against the (a) Ti-6Al-4V and (b) Co-Cr alloys.

4. Conclusion

In conclusion, the Ti-6Al-4V alloy showed relatively poor tribocorrosion performance and caused heavy surface damage on the alumina counter ball with increasing the coefficient of friction. The wear debris generated from Ti-6Al-4V alloy led to an increase optical density of the SAS solution along with heavy fluctuations in the OCP potential and friction coefficient values through out testing period compared to the Co-Cr alloy. However, considering the implants applications, the Ti-6Al-4V alloy was found to be the best candidate with relatively low elastic modulus and outstanding biocompatibility in comparison to Co-Cr alloy. Thus, surface modifications of this alloy for the load bearing applications are very important for achieving further developed biocompatibility and the second removal surgical operation can be avoided.

Acknowledgements

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TRIBO-CORROSION BEHAVIOR OF CAST AND SELECTIVE LASER MELTED Co-Cr ALLOY FOR DENTAL APPLICATIONS

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Abstract: Cobalt-chromium based alloys are widely used in dental applications due to their excellent mechanical properties, high corrosion resistance and good biocompatibility. Although they are generally fabricated by casting for dental restorations, recently selective laser melting (SLM) has become an attractive production method since it allows complex geometries. Recent studies revealed that Co-Cr alloys formed by SLM, provided better corrosion resistance as well.

In this study, tribo-corrosion behavior of a Co-Cr-Mo alloy produced by casting (Biosil-Degudent) was compared with the one (Co212-f ASTM F75) produced by SLM. The wear properties were investigated by tribo-corrosion tests in a Fusayama-Meyer artificial saliva solution using a “ball-on-disc” type tribometer. Polished surfaces were tested against zirconia balls at the same sliding speed, distance and load. Friction coefficient values were determined and the worn surfaces were evaluated using microscopes to compare the wear and tribo-corrosion resistance through wear tracks. SLM proved to be a promising manufacturing method for dental applications.

Keywords: DENTAL Co-Cr ALLOYS, CASTING, SELECTIVE LASER MELTING, TRIBO-CORROSION

1. Introduction

Due to a great demand in artificial hip, knee joints and dental applications, many attempts were made to produce materials having superior physical and chemical properties [1]. Co-Cr-Mo alloys are commonly used as implant materials as well as restoration materials in dentistry due to their excellent mechanical properties, high corrosion resistance and good biocompatibility [2]. They possess face centered cubic (FCC) lattice - γ phase at high temperatures and hexagonal close packed (HCP) lattice - ϵ phase at room temperature [3-6]. While γ phase is responsible for ductility, ϵ phase enhances the corrosion and wear resistance [7]. In proper alloying the microstructure of the dental alloys is composed mainly of γ phase and carbides of the $M_{23}C_6$ type [4]. Chromium, molybdenum and tungsten are added for strengthening of the solid solution. The chromium (22%-28%) forms a passive oxide layer on the surface providing good corrosion resistance and carbides in the microstructure increase the hardness and wear resistance [4, 6]. Consecutively, the properties of the dental alloys depend on the γ - ϵ ratio and the type, quantity and distribution of the carbide phase in the microstructure.

Not only grade of material but also processing technique have an important role on the final properties of implant materials [8]. From past to present, casting is a well-known technique to produce several metallic products, however, cast alloys having inhomogeneous microstructure, solidification defects, segregation and large grains are not sufficient to ensure high quality, superior physical and chemical properties leading to a long service life [9]. Today, selective laser melting (SLM) technique, a kind of additive manufacturing method, is preferred in fabricating implant materials due to its highly motivated facilities [10]. In this technology layers of metal powder are fused into a 3D model by adopting a computer-directed laser [11-13]. The advantages of SLM over the traditional methods include production of personalized complex objects; manufacturing of parts with dense structure and predetermined surface roughness; controllable, easy and relatively quick process [11]. The SLM process is characterized by high heating and cooling rates of the melted layer as well as heating and solid state phase transformations in the underneath layers, which determine microstructure and properties quite different than that of the cast details.

It was established by Meacock et al. [14] that the microstructure of biomedical Co-Cr-Mo alloy, produced by laser powder microdeposition, is homogenous comprised of fine cellular

dendrites. The average hardness was 460 HV0.2, which is higher than the values obtained by the other fabrication processes. Barucca et al. [15] investigated Co-Cr-Mo parts, produced by direct metal laser sintering. They established that the higher hardness is attributed to the presence of the ϵ -lamellae grown on the $\{111\}\gamma$ plane. Dolgov et al. [16] also reported about more homogeneous microstructure, the higher hardness and yield strength of the SLM fabricated Co-Cr-Mo alloy (382 HV and $R_{0.2}$ =720 MPa) comparing to the cast alloy (335 HV and $R_{0.2}$ =410 MPa). Lu et al. [17] investigated the microstructure, hardness, mechanical properties, electrochemical behavior and metal release of Co-Cr-W alloy fabricated by SLM in two different scanning strategies – line and island. They established the coexistence of the γ - and ϵ -phases in the microstructure and nearly the same hardness (570 HV for line-formed alloy and 564 HV for island-formed) as well as high corrosion resistance in the both production schemes. The higher hardness and the more homogeneous microstructure of SLM Co-Cr alloys are preconditions for higher wear and corrosion resistance, respectively.

Tribo-corrosion is a relatively new field of physical science research in which two degradation processes, mechanical wear and electrochemical responses to particular consequences of that wear, are studied [18]. In general, the tribo-corrosion behavior of metals can be attributed to the synergy between wear and corrosion and the leading phenomena can be summarized as the following: (i) an oxide film forms on the metal surface and it protects the surface from corrosion degradation, (ii) wear removes the film inducing an accelerated corrosion process, (iii) wear-corrosion products enhance the wear, (iv) the synergy between wear and corrosion exponentially causes a great material loss. This synergy can be varied by not only test conditions (corrosive medium, temperature, tribological pairs, load, sliding speed etc.) but also microstructural features [19]. In this study, cast and SLM processed two commercial alloys were tested in artificial saliva solution to understand their tribo-corrosion behavior throughout the microstructural features.

2. Materials and methods

Materials and sample preparation

In this study, two commercial Co-Cr-Mo alloys (Biosil-Degudent and Co212-f ASTM F75) were selected as test materials. Biosil-Degudent alloy was produced by casting and its composition includes 64.80 Co - 28.50 Cr - 5.30 Mo - 0.50 Si - 0.50 Mn - 0.40 C,

wt-%. It has a hardness value of 400 HV10 and its yield strength and tensile strength are 700 MPa and 900 MPa, respectively. The chemical composition of SLM processed Co212-f ASTM F75 alloy contains 65.20 Co - 28.30 Cr - 5.48 Mo - 0.754 Si - 0.164 Fe - 0.036 V, wt-%. Its hardness value is 35 HRC and yield and tensile strength values are 835 MPa and 1050 MPa, respectively. All data about the chemical composition and also mechanical properties are provided by the manufacturer.

Test specimens with dimensions 2 mm x 10 mm x 40 mm were prepared by lost-wax casting and SLM process. At first a silicone mold for manufacturing of wax models for the cast test specimens was fabricated, while the 3D model was created with SolidWorks software. The cast samples were produced by centrifugal casting of Co-Cr alloy “Biosil”. The SLM samples were fabricated directly from the virtual 3D models using SLM 125 machine of the “SLM Solutions”, Germany. The machine is equipped with continuous Nd:YAG laser which worked with power 100 W and laser spot diameter 0.2 mm. The metal powder of Co-Cr alloy Co212-f ASTM F75 was melted in layers with 0.03 mm thickness until the desired construction was obtained. During manufacturing process the laser at first scanned the outer contour of the layer of the first specimen’s part, next it hatched the area within the boundaries at an angle of 45° with a pitch of 0.13 mm. After that it passed to the same layer of the next specimen’s part, thus fabricating the whole layer. The SLM technological regime, recommended from the company producer was used.

Sample characterization

Samples were cold molded and surfaces were prepared by standard metallographical techniques, namely grinding with SiC papers, polishing with diamond paste. Surfaces were etched electrolytically in a solution (5 % HCl, 95 % H₂O), at 5 V for 1 sec. Microstructural characterization was carried out using light microscope (LM, Olympus BX41M-LED) and scanning electron microscope (SEM, Jeol JSM 6060) equipped with energy dispersive spectrometer (EDS, IXRF).

Tribo-corrosion test

For tribological studies, a “ball-on-disc” type tribometer (Nanovea Microtest SMT/A) was used and the system was supported by a home-made attachment for the tribo-corrosion tests. In this system (Fig. 1), molded alloys having equal contact areas were fixed into a polymeric chamber and polished surfaces were subjected to a Fusayama-Meyer artificial saliva solution (pH ~ 7.1). The counterpart material was selected as ZrO₂ having 3 mm diameter and the tests were carried out under 5N normal load, at a speed of 0.01 m/s during 100 m for 3 ½ h.

All test results were evaluated by (i) variation of friction coefficient (COF) as a function of sliding distance, (ii) determination the change in weight, (iii) worn surface examinations by SEM. Weight loss values for all samples were determined by weighing the specimens before and after the tests using a precise laboratory type balance.

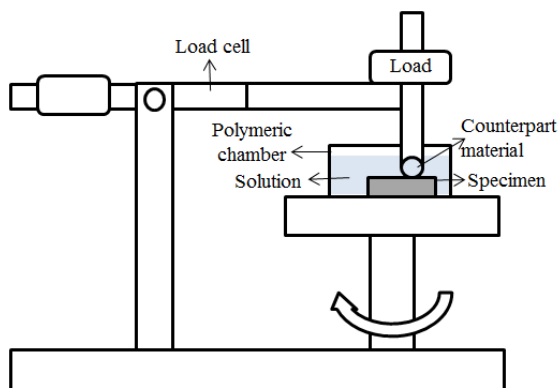


Fig. 1: A schematic illustration of a “ball-on-disc” type tribometer equipped with tribo-corrosion unit.

3. Results and discussion

Fig. 2a shows a typical cast structure of Co-Cr-Mo alloy and it consists of γ -Co dendritic matrix of face centered cubic structure and a secondary blocky $M_{23}C_6$ carbide phase segregated to the columnar grained structure (Fig. 2b). As reported in several studies [4, 16, 20-22], the cast matrix includes large grains, non-homogenous microstructure, solidification defects resulting in poor mechanical properties. The coarse grain structure will be very effective on the chipping failure thus the material used in dental applications should consist of smaller grains [17]. This problem could be overcome by SLM which is an additive manufacturing technique, fabricating metal products having very fine and well distributed microstructural features within matrix [14-17, 20]. In SLM processed materials, laser scan produces a layer-layer molten pool boundaries (MPBs) given in Fig. 3a and SEM observations at higher magnification revealed out the presence of an extremely fine microstructure inside a single pool (Fig. 3b) and very fine carbides having globular (drawn by circle) and lamellar (drawn by square) morphology are observed in re-solidified structure (Fig. 4).

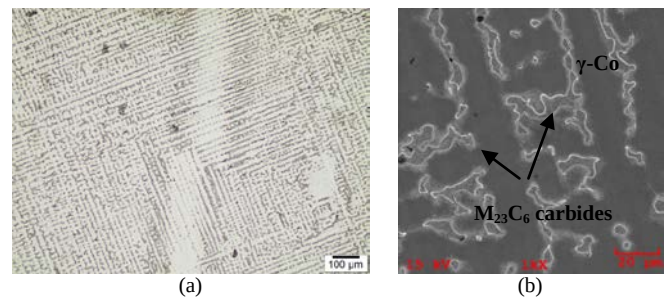


Fig. 2: Microstructures of cast Co-Cr-Mo alloy: (a) LM image showing typical cast structure γ -Co dendritic matrix, (b) SEM image showing secondary $M_{23}C_6$ carbides at the boundaries.

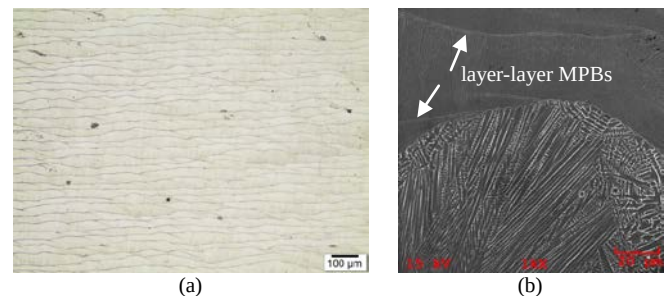


Fig. 3: Microstructures of SLM processed Co-Cr-Mo alloy: (a) LM image showing layer-layer MPBs, (b) SEM image revealing out the finer structure compared to cast alloy.

Fig. 5 shows the variation of COF as a function of sliding distance for tested alloys. The curves include typical fluctuations due to ploughing and adhesion of moving particles formed by wear and corrosion and they consist of two distinct regimes: (i) running-in regime at first 10 m and (ii) steady-state regime after 25 m. Till the end of running-in regime, the cast alloy has a tendency to increase its COF value and it has a peak value of 0.09, however, COF value of SLM alloy initially has its maximum value as 0.13 and then goes down during sliding. At the steady-stage regime, the COF values of both alloys have no significant change and vary in the range of 0.08 – 0.10. Studied alloys owe their wear resistance to the hard carbides (especially Cr-rich carbides) within their matrix, however, these carbides have a detrimental effect in corrosive medium due to depletion of chromium away from the matrix and lack of protective film [predominantly Cr₂O₃ and Cr(OH)₃] on the metal surface [23]. This detrimental effect can be observed as pits and crevices in the surrounding areas of the carbide due to localized attack in corrosive medium. In sliding of tribological pairs in a corrosive medium, the corrosion resistant carbides stick out and behave as asperities deteriorating the protective film, grooving the surface, leading to corrosion products and increasing the COF [6]. All these well-known approaches make it clear why the cast alloy having coarser microstructural features (columnar structure and

blocky carbides) than that of SLM processed alloy exhibited a rapid COF increase in running-in regime. On the other hand, SLM processed alloy keeps its chromium in the matrix, therefore, more stable oxide layer makes the alloy resistant to both corrosion and wear despite a normal force is subjected to tested alloy embedded into a corrosive medium and corrosion potential shifts from a passive region to a more active region due to frictional forces.

As mentioned before, the synergy between wear and corrosion causes a great material loss forming an adhesion layer. The presence of adhesion layer may change the tribological interaction from two-body contact to three-body contact at the steady-state regime in which both cast and SLM alloys exhibit similar COF values. Not only variation of COF values but also changes in weight support the effect of microstructural features of Co-Cr-Mo alloys on tribo-corrosion behavior. When the weights of the alloys before and after the tribo-corrosion test were considered, a gain of 3.3 mg and 1.8 mg were observed for the cast and SLM alloys, respectively. The increase is mainly due to the corrosion products accumulated on the worn surfaces together with the wear debris, and it is almost twice as much for the cast alloy compared to the SLM one clearly indicating that it suffered more from corrosion and wear under the given conditions.

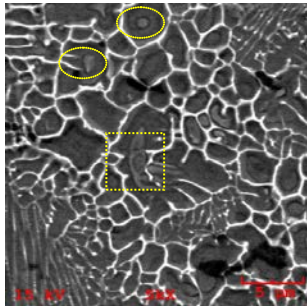


Fig. 4: SEM image showing globular and lamellar shaped carbides in columnar structure of SLM processed alloy.

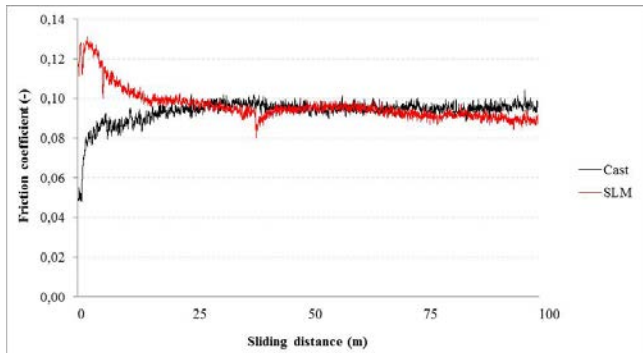


Fig. 5: Variation of COF values as a function of sliding distance.

The worn surfaces of studied alloys were also examined by SEM and Fig. 6 shows the general views of wear tracks formed on the surfaces. Worn surface of cast alloy exhibited a very smooth wear track and a thick degraded film layer compared to that of SLM processed alloy. The film layer formed on cast alloy consists of 21.32 O - 0.20 Na - 0.70 Si - 6.90 P - 0.01 Cl - 4.32 Ca - 18.30 Cr - 10.36 Fe - 5.20 Mo - 32.69 Co (wt-%) and the elemental distribution indicates that (i) the degraded film includes not only passive layer of Cr-Co-Mo oxides like Cr_2O_3 , CoO and MoO_3 but also Fe and Si based oxides due to depletion of Fe and Si ions having more negative standard reduction potential close to that of Cr, (ii) the presence of Cl and Na is a good evidence for the formation of chloride salts, (iii) the presence of phosphorus and calcium is related with the formation of a phosphate and carbonate compounds as reported in previous studies [23]. The film layer of SLM processed alloy has also similar elemental distribution (25.55 O - 0.43 Na - 0.63 Si - 6.45 P - 0.04 Cl - 3.00 Ca - 20.70 Cr - 8.42 Fe - 4.35 Mo - 30.43 Co, wt-%) on its corroded surface.

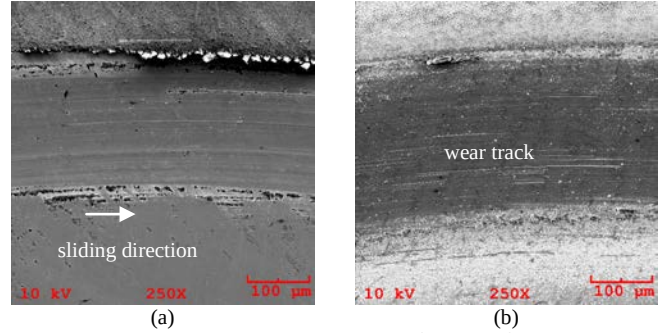


Fig. 6: SEM images showing a general view of worn surfaces: (a) cast and (b) SLM processed alloy.

SEM image given in Fig. 7 shows the pittings and cracks on the worn surface of cast alloy. In sliding conditions, the removed particles can be cold welded and then layered due to adhesive forces and then adhered layer can be deteriorated by delamination during contact. In this case, the detached particles will be very effective in wear and form new surfaces for the friction within the frame of three-body contact. Meanwhile, pitting corrosion is involved as point or small hole on the surface of metal embedded into a corrosive medium and pits can initiate at surface defects. A rapid depletion of oxygen in the pits causes metal oxidation and a progressive damage consisting of crack initiation and propagation into the porous and brittle oxide film starts [24, 25].

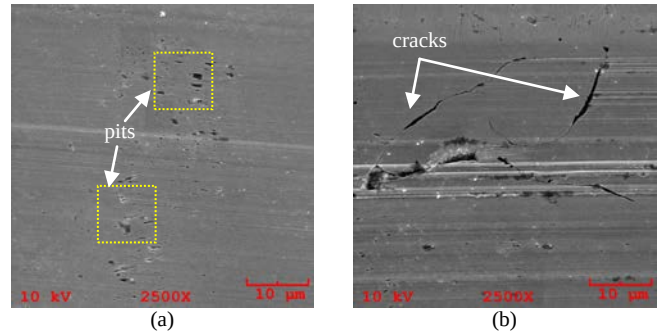


Fig. 7: SEM images showing several pits (a) and (b) cracking in adhered layer on the worn surface of tested cast alloy.

The worn surfaces given in Fig. 8 exhibited very different surface characteristics compared to cast alloy. The surface has very sharp plastic flows which are oriented step by step due to layer-layer structure of SLM processed alloy and also deep grooves indicating abrasion type wear. As examined before, SLM processed alloy has a finer microstructure compared to cast alloy and this structure makes it harder by plastic deformation during the contact of a harder counterpart. A decrease in the hardness gradient between tribological pairs increase the scratch capability of any surface and the abrasion wear appears.

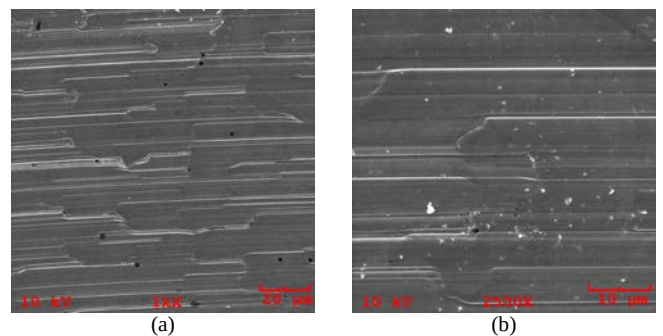


Fig. 8: SEM images showing plastic flows on the worn surface of SLM processed alloy: (a) plastic flows which are oriented step by step due to layer-layer structure of tested alloy, (b) deep grooves indicating the abrasion type wear under studied conditions.

4. Conclusions

The wear properties of Co-Cr-Mo alloys, fabricated by casting and SLM, were investigated by tribo-corrosion tests in a Fusayama-Meyer artificial saliva solution. As a result of different production processes, the microstructures of the cast and SLM samples differ considerably, thus revealing different wear and corrosion properties.

The friction coefficient (COF) of the cast alloy has a tendency to increase till the end of the running-in regime and it has a peak value of 0.09. COF value of SLM alloy initially has its maximum value as 0.13 and then goes down during sliding. At the steady-stage regime, the COF values of both alloys have no significant change and vary in the range of 0.08 – 0.10.

The examination of the worn surfaces reveals different wear types for the cast and SLM Co-Cr-Mo alloys. The worn surface of the cast alloy exhibits a very smooth wear track with many pits and cracks as well as a thick degraded film layer. While the worn surface of the SLM alloy has very sharp plastic flows which are oriented step by step due to the alloy's layer-layer structure and also deep grooves exist indicating abrasion type wear.

Due to the more homogeneous microstructure, the higher hardness and the more stable oxide layer formed on the surface, the SLM processed alloy is more resistant to both corrosion and wear, thus proving that the SLM is a promising manufacturing method for dental applications.

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OBTAINING OF GAS-TIGHT GLASS-CERAMIC JOINTS OF MULLITE ARTICLES THROUGH THE NANOCRYSTALLIZATION OF Y_2O_3 - Al_2O_3 - SiO_2 GLASS

ИЗГОТОВЛЕНИЕ ГАЗОПЛОТНЫХ СТЕКЛО-КЕРАМИЧЕСКИХ СОЕДИНЕНИЙ ДЕТАЛЕЙ ИЗ МУЛЛИТА ПУТЕМ НАНОКРИСТАЛЛИЗАЦИИ Y_2O_3 - Al_2O_3 - SiO_2 СТЕКЛА

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Abstract: Obtaining gas-tight joints between mullite articles was made by crystallization of yttrium aluminosilicate glass. The glass joint layer softens at joining temperature (1450-1470°C) to bond the mullite samples without additional pressure and then crystallized during cooling. The good adhesion of crystallized glass to mullite surfaces ensure the gas-tight joints with permittivity $2.5 \cdot 10^{-4}$ sccm/cm. The eutectic like crystallization of initial yttrium aluminosilicate glass into yttrium silicate and mullite lead to formation the nanocomposite structure which ensure the 4 point bending strength value near 105 MPa.

Keywords: GAS-TIGHT JOINTS, MULLITE, GLASS, CRYSTALLIZATION

1. Introduction

Mullite $3Al_2O_3 \cdot 2SiO_2$ is a good and cheap oxide material for high temperature structural and refractory applications because of high enough mechanical properties at high temperatures and acceptable thermal conductivity value (6-15 W/m·K). However, fabrications of complex form details from ceramic materials are limited by ceramic brittleness, increasing of machining cost and material losses from cutting procedure. So, the assembling of complex shapes from simple parts by joining can be acceptable procedure. For some applications, for example SOFC, the joining procedure is a basic problem in the stack fabrication procedure. The high temperature strength, creep and gas impermeability of joints are the key factors in case of devices, which works under gas flows and mechanical loads.

It is well known the several methods of the joining of ceramic materials: i) joining by brazing technique (Ag, Au, etc.) [1]; ii) joining by mechanical load – sealing [2, 3]; iii) joining by glass-ceramic intermediate layer [4, 5, 8]. The brazing technique is often used in electronic industry and vacuum technique and its application limited of 400-500°C working temperatures. The sealing technique involves the use of an external load for seam sealing. The glass-ceramic interlayer joins can work at high temperatures (800-1000°C) and maintains their properties for a long time.

The good joining with glass-ceramic interlayer is ensuring by the good wettability and low mismatch of thermal expansion coefficients between the interlayer and the mullite. Besides, the low level of viscosity (near 10^5 dPa·s) should be in glass state for ensure the wetting of ceramic material and high level of viscosity (near 10^9 dPa·s) should be in glass-ceramic state for prevention of outflow of joint material from seam. In view of these requirements, Y_2O_3 - Al_2O_3 - SiO_2 glasses (YAS) seem to be the suitable joint material for the joining of mullite material.

In this research, we study the structure formation, strength and gas permittivity of joint between the mullite material (bars and pipes) with yttria–alumina–silicate (SiO_2 54 wt%, Al_2O_3 18 wt%, Y_2O_3 28 wt%) glass powder. The optimal joining conditions, microstructures and strengths of the joint, as well as gas permittivity, were characterized.

2. Experimental

2.1. Material preparation

Mullite samples were fabricated by sintering of mullite powder, which was synthesized by calcination of the mix of the aluminum hydroxide with silica hydroxide in stoichiometric composition. The $Si(OH)_4$ or hydrous silica gel and aluminum hydroxide were precipitated in ammonia water solution separately. The wet silica gel and aluminum hydroxide were mixed in water, filtered, dried in

a microwave furnace with an output power of 700 W at a frequency of 2.45 GHz and calcined in a resistive furnace at 1200°C for obtaining maximal value of mullite phase [6, 7].

For determination of mechanical properties of sintered mullite materials and joined samples the rectangular shape samples (40*5*20 mm and 25*25*1 mm) and tube with diameter 20 mm, height 30 mm and wall 3 mm thick were prepared firstly by means of uniaxial cold pressing at 60 MPa, then isostatic pressing at 200 MPa and finally pressureless sintering at 1600 °C for 1h.

The composition of YAS glass was SiO_2 54 wt%, Al_2O_3 18 wt%, Y_2O_3 28 wt%. This composition has very high melting temperature (near 1650°C) and we melted the oxide mix at 1800°C 30 min in inductive furnace. After that the glass was quenched in water and ball milled in planetary mill MSK-SFM-1 (MTI Corp., USA) at 400 rpm for 4 h using YSZ milling media.

2.2. Materials characterization

The powders, sintered specimens and crystallized YAS glass-ceramic were characterized by means of XRD (Dron-3) with Cu-K α radiation for crystallite sizes and quantitative phase analyses. Particle sizes of different calcined powders were estimated by means of transmission electron microscopy (JEM 200, Jeol, Japan). The microstructures of the mullite ceramics and its joints were studied by scanning electron microscopy (JSM 6490LV Jeol). The density of sintered mullite material was determined by Archimedes method.

The shrinkage data of the pressed pellet from this glass powder was obtained using a dilatometer (DIL 402 PC, Netzsch) during heating sample till melting process begun. The dilatometer was calibrated using a standard sample of Al_2O_3 . The detected control points during heating the glass sample T_d – 958.5°C, T_c – 1368°C and T_m – 1420°C (Fig. 1) were in a good agreement with literature data.

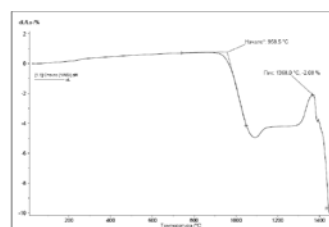


Fig.1 The shrinkage curves for YAS glass powder

The joints between two (40*20*5mm) mullite bars were carried out on 40*5 mm side by YAS glass powder slurry (Fig. 2). The glass powder slurry was prepared by mixing glass powder in 3wt% polyvinilbutiral ethyl alcohol solution. The joined samples were cut perpendicular the joint seam for obtaining rectangular samples

5*5*40 mm for testing on 4 point bending. The flexural strength was measured using a four-point bending test on polished samples with a cross-head speed of 0.5 mm/min (Tinius Olsen H50kT, USA).

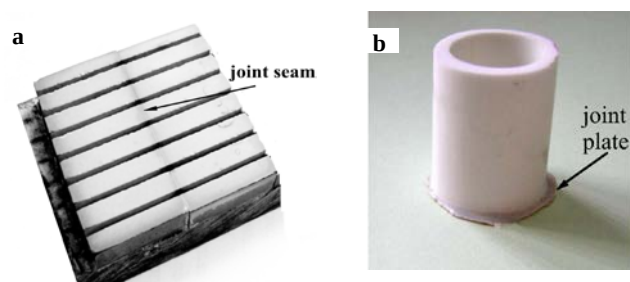


Fig. 2 The joined mullite samples for bending strength (a) and gas permittivity (b) testing

For gas tight test the joints between sintered mullite tube and plate, which were cut from bar (25*25*1 mm) were prepared. The joint regime was: heating to 1470°C with 180°C/h, dwelling time 2h and heating to 1200 with 120°C/h and final cooling to room temperature with a furnace. Measuring of joints gas permittivity was carried out on monitoring of pressure in the joined tube samples by DDR-1200 - pressure monitor (J-KEM Scientific, USA), which can support and measure pressure in the set volume. The measuring provided at next regime: heating to 100, 200, ...900°C at 150°C/h, exposure on temperature and measure pressure decay, heating at next temperature and so on. The measurement system of pipes, valves and other (without sample), has leak $1.5\text{--}2.2 \cdot 10^{-6}$ Pa*m³/c at pressure in the system of 40-50 kPa. The measurement of test-tube sample or closed sample (with identical volume) shows that leak of test-tube sample and system was $2.2\text{--}2.7 \cdot 10^{-5}$ Pa*m³/c.

3. Results and discussion

The XRD data shown that after calcinations at 1200°C the formation of mullite powder is almost completed (92% mullite and 6% cristobalite). After sintering of this powder at 1600°C the phase composition was represent only mullite phase. The chemical composition of powders and sintered samples were analyzed by SEM with EDS analysis. The chemical composition of mullite was practically corresponding to stoichiometric composition $\text{Al}_2\text{O}_3\text{:SiO}_2$ as 72.5:27.5. The density value of sintered mullite material was 3.11 g/cm³, porosity – 0.35%. The SEM investigations (Fig. 3) of fractured surface represent a uniform distribution of micron size of mullite grains (average diameter 2.8 μm). The coefficient of thermal expansion (CTE) of mullite material was $6.28 \cdot 10^{-6}$ /K. At the sintered and cooled YAS sample the CTE was also measured. In temperature region 200 – 900°C the CTE value was $6.7 \cdot 10^{-6}$ /K.

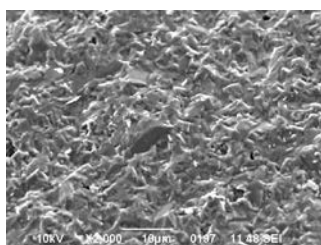


Fig. 3 SEM image of fracture surface of sintered at 1600°C mullite material

The 4 point bending strength value of mullite material was 184 ± 22 MPa and for joined samples - 104 ± 16 MPa. This value in several times higher in comparison with strength value which can be obtained under joining by noncrystallized glasses.

The testing of joined sample on gas tightness shows that the in temperature range from RT to 900°C the leak rate through the joining made from YAS glass between tube and plate from mullite material does not exceed $2.5 \pm 0.5 \cdot 10^{-4}$ sccm/cm (Fig. 4). This is high enough values, which can be compared with tightness of SOFC components [8].

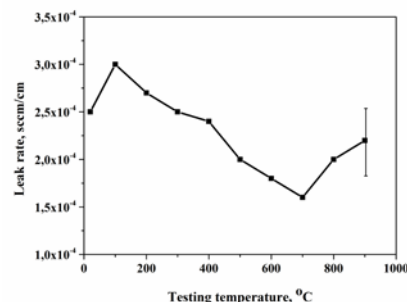


Fig. 4 Leak rate versus testing temperature through joints between two mullite samples joined by YAS glass

As is well known the strength values of glass materials is very low and can not ensure the strength of joints. Increasing of strength value of glass material achieves by addition in glass material ceramic or metallic particles or carried out the partial crystallization lead to formation oxide inclusions in a glass matrix. The crystallization of YAS glass is typical example of such strengthening. The advantage of YAS glass in mullite joining process is the formation of mullite phase during crystallization.

The YAS glass was investigated after joining process (heating and cooling) for determination of crystallization behavior and changing of chemical composition. The joint layer thickness was near 70 μm. The joint layer and border between ceramic mullite material and joint material do not contained the porosity. This is evidence that joint regime is correct. After crystallization the joint material consists basically from yttrium silicate and mullite phase. At SEM image we can see the character needle like crystals of mullite (Fig. 5). The small amount of third phase (white crystals) was not identified. The concentration of alumina in these inclusions is slightly decreased (3-5%) (spectra 5, 6, 7 in Table 1) and yttrium oxide increased in comparison with average values (spectrum 3 in Table 1).

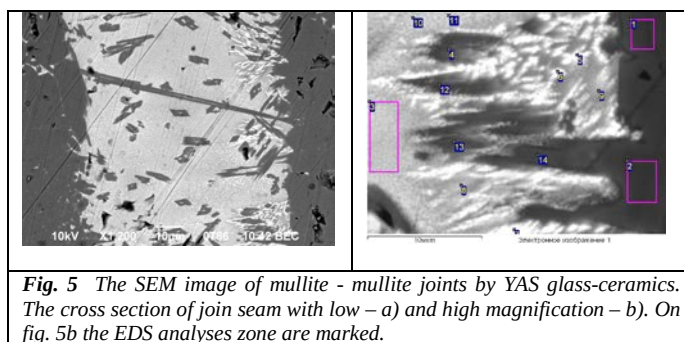


Fig. 5 The SEM image of mullite - mullite joints by YAS glass-ceramics. The cross section of joint seam with low – a) and high magnification – b). On fig. 5b the EDS analyses zone are marked.

Table 1. The EDS analysis data of crystallized YAS glass. The number of spectrum corresponds to analyzed zones on fig. 5b.

Spectrum	Concentration, wt%		
	Al_2O_3	SiO_2	Y_2O_3
1	72.40	27.60	0
2	72.79	27.21	0
3	18.03	51.61	30.37
4	30.28	58.05	11.67
5	13.75	49.56	36.68
6	15.83	51.02	33.15
7	14.59	48.83	36.58

At bigger magnification we see that crystallization in YAS glass at cooling pass by eutectic like mechanism (Fig. 6). The small points of darker phase – the mullite crystals in yttrium silicate glass-ceramics. A typical inclusions mullite size less than 20 nm.

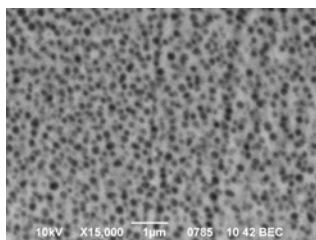


Fig. 6 SEM image of joint layer from crystallized YAS glass

So, the formation of composite or nanocomposite structure during crystallization YAS glass lead to increasing the strength value of joint seam between two mullite samples. The good adhesion of YAS glass to mullite material and low CTE mismatch between mullite and YAS glass-ceramic allow producing the gas-tight joints. In addition, this method of the joining by YAS glass-ceramic can be used to join for other materials with close CTE values, for example SiC.

Conclusion

Yttrium aluminosilicate glass powder was used as the joint material for joining two mullite samples of different forms. The glass joint layer softens at joining temperature (1450-1470°C) to bond the mullite samples without additional pressure and then crystallized during cooling. The good adhesion of crystallized glass to mullite surfaces ensure the gas-tight joints with permittivity $2.5 \cdot 10^{-4}$ sccm/cm. The eutectic like crystallization of YAS glass into yttrium silicate and mullite lead to formation the nanocomposite structure which ensure the 4 point bending strength value near 105 MPa. The YAS glass can be used as joint material for bonding materials with CTE value near $6 \cdot 10^{-6}/K$.

Acknowledgments

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