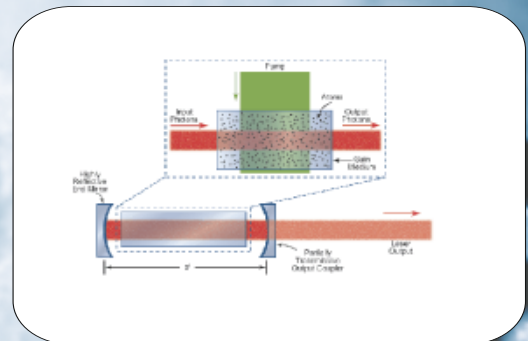


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C O N T E N T S

MACHINES

Methods of Identifying Air Leaks in Pneumatically Operated Equipment in the Industry Adriana Kamenszká 1 Miriam Matúšová	207
--	-----

TECHNOLOGIES

The influence of electric discharge and solutions of surfactants on the filtration characteristics of porous, liquid-saturated media Olha Syzonenko, Ivan Belinskyi, Ivan Denisyuk, Andrii Torpakov	212
Manufacturing Principals of Metal processing Lasers Nikolaos Papageorgiou	216
Determination of the quality of welded joints after repairs of hot water pipes Ján Viňáš	219
Effect of production technique on a high silicon al-si-ni sintered alloy structure and properties Kaverynskyi V., Bagliuk G., Verbilo D. G., Kyrylyuk S. F., Sukhenko Z., Rud A. D.	224
Development of Software Application in the Evaluation of Erythrocyte Aggregation Studied with Microfluidic Device Emilia Abadjieva, Anika Alexandrova, Tihomir Tiankov, Ina Giosheva, Svetla Todiniva	228

MATERIALS

Polymer blends based on regenerated thermoplastics with low flammability Alexander Antonov, Pavel Klochko, Vasily Struk, Nikolay Prokopchuk, Alexander Lesun, Wan Xuemin	232
Size distribution of the shape memory alloys powders obtained by pulsed electrical discharge in cryogenic liquids Gennady E. Monastyrsky	236
Titanium-Based Porous Structures Produced by Powder Metallurgy Approach Oleksandr O. Stasiuk, Denys V. Oryshych, Dmytro V. Pakula, Vasyl I. Nevmerzhytskyi, Vasyl P. Tkachuk	240

Methods of Identifying Air Leaks in Pneumatically Operated Equipment in the Industry

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Abstract: In recent decades, it has become commonplace to automate assembly operations, often linked to production technologies within a single flexible production and assembly system. Assembly has a significant impact on global production, both in terms of its share of the total production cost of assembled products and in terms of the number of people employed in this area. The challenge for scientific and technical intelligence is to rapidly acquire production and assembly know-how to achieve target parameters in the shortest possible time. The use of all machinery and equipment forces people to think about how to use energy in the most efficient way, with the least possible loss, either in terms of money or in terms of energy. Every year the cost of energy increases, whether due to depletion of supplies or climate changes. Compressed air leaks are a serious problem in many industrial and manufacturing environments. These leaks not only increase operating costs but can also have a negative impact on the environment and the health of workers. Compressed air is the most expensive medium and when all basic costs are included, compressed air is many times more expensive than the equivalent of electricity. Therefore, it is necessary to analyse how to prevent high losses in the industry.

Keywords: PRODUCTION AND ASSEMBLY PROCESS, PRESSURE, AIR, ASSEMBLY, LEAKS, FAULT IDENTIFICATION

1. Introduction

The impact of complex automation is also influencing the development of engineering. Today, complex automation is the basis of almost every production and assembly system. Industrial production is currently at the crossroads of two development stages. The stage that was a huge boom is coming to an end as computers and also electronic and information systems control industries. However, into this automation were also involved humans who have had to program logic drivers, robots and the rest. The new industrial revolution is referred to as the era of digitalization, in which industries are being transformed by intelligent machines, data collection processes and artificial intelligence that can make autonomous decisions in real-time.

The impact of such developments in production and assembly technology is leading companies around the world to increasingly focus on flexible automation. Such a system includes technological workstations, which in many cases are fully automated handling systems, transport systems, storage systems, control systems and other auxiliary equipment. In recent decades, it has become commonplace to automate assembly operations, often linked to production technologies, within a single flexible production and assembly system. The functionality of the entire production and assembly system must be designed, tracked and set up for efficient production. Component wear or system unreliability can cause losses that need to be tracked and then eliminated. If air is used as the medium for the work process of the assets within the production and assembly systems, this medium must be monitored and recorded for potential losses and leaks.

2. Process of Design and Manufacture of Production and Assembly Line

When designing and manufacturing production and assembly lines, it is important to have a clear idea of the process that the production and assembly line will go through.

A process is defined as a logical sequence of activities and steps that are designed and implemented to achieve a specific output or result. A process in an enterprise has a clearly defined beginning, flow and end, with linkages to other processes. It is important to analyse the outputs of a process and ensure that the inputs and outputs between processes are identical. Each process has a 'process card' which provides detailed information on the process name, process operator, inputs, outputs, previous processes, following processes, resources, etc. Defining the objectives and measurable indicators of the process are key to its successful implementation and monitoring of results. The resources required to implement the processes should be used optimally to achieve maximum process efficiency. Process mapping is an important element for a better understanding and significant improvement of processes [1, 2].

2.1 Process Mapping

Process mapping is a proven analytical and communication tool designed to optimize current processes and introduce a new process-oriented company structure.

A process map (Figure 1), also known as a flow diagram, is a tool for visualising processes and their individual steps. A process map starts by identifying the inputs and outputs of a process. Inputs may include raw materials, labour, financial resources, etc., while outputs may be finished products, semi-finished products, or other output products of the process. Thereafter, the sequence of steps required to achieve the process objectives is mapped. These steps include the individual workstations and machines that are part of the production process. The process map includes information about the time requirements that are associated with each step of the process and also information about the responsibilities for each step [3, 4, 5].

2.2 Project management of the design and production of a production and assembly line

When designing and setting up a production and assembly line, the line goes through a large number of different processes that build on each other. Project management is a process aimed at managing the acquisition of projects and the development of processes and products to ensure efficient industrialisation.

To incorporate a process, the following information must be defined:

- **Previous project management processes** - e.g. strategic planning, marketing, management of business activities.
- **Inputs** - customer documentation, request for quotation, customer requirements.
- **Outputs** - the product that meets customer requirements, optimised project profitability.
- **Subsequent processes** - maintenance of the line, customer satisfaction assessment, management of business activities.
- **Resources** - staff, infrastructure, hardware, software [6, 7].

2.3 Conceptual Description of the Steps in the Design and Construction of the Production and Assembly Line

At the beginning of each design of a new production and assembly line, the customer places an order. All available design documentation provided by the customer must be reviewed by the project manager. If the project is feasible, the order is confirmed. After these initial steps, meetings are convened to discuss and agree on the technical specifications of the production and assembly line, both internally and externally with the customer.

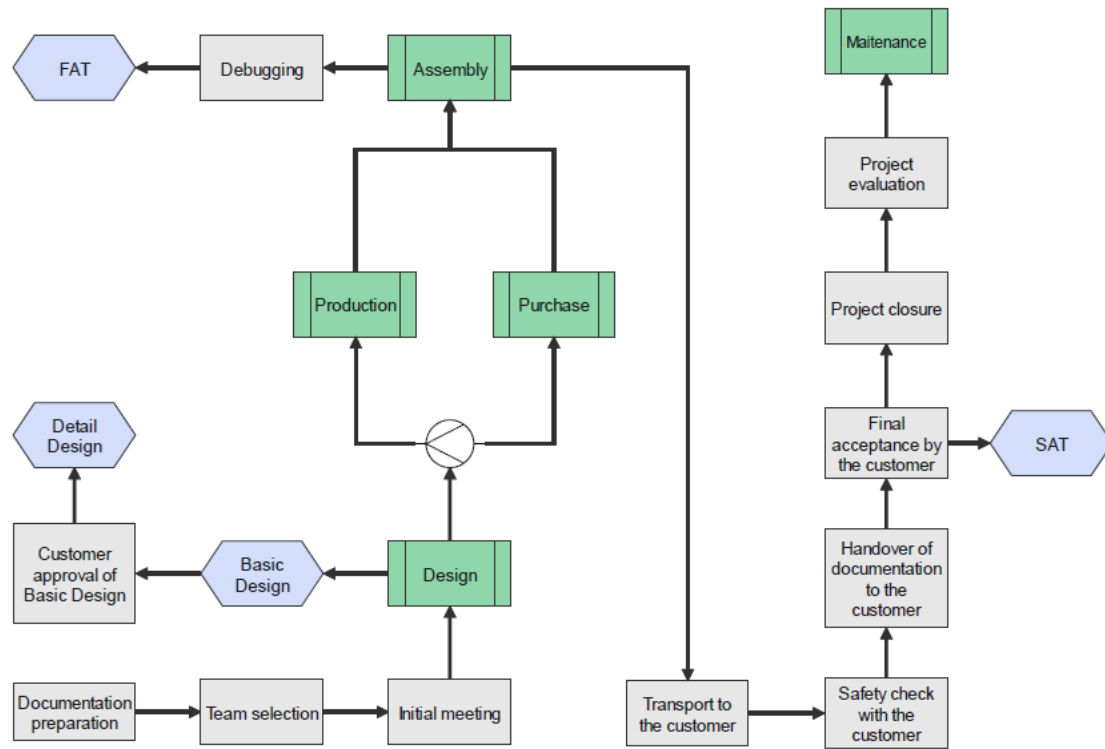


Fig. 1 Flow diagram of processes and steps

The integration of people into the team by the heads of each department for a specific project occurs as soon as the order is approved by the project manager together with the customer. Once the team is formed, an initial meeting is called where the requirements and details of the future production and assembly line are discussed for the first time within the team. After the meeting, various management plans are prepared. At each point and at each stage of the project, a management plan and a register of potential risks must also be prepared.

This moves the project into the design phase. The project manager is tasked with communicating with the client and then relaying information from the client to specific departments. The design department is tasked with delivering the brief in all its aspects, within the specified time and budget. It also has the task of communicating with the client and submitting proposals at specified intervals. The basic design phase includes the initial conceptual and functional design of the production and assembly line. After incorporating any changes requested by the customer, the basic design of the project is submitted to the customer for approval. This is the design freeze and the detailed design is implemented in the next phase. When the detailed design is created, the material budget is also checked for compliance. The final stage of the design, after customer approval, is the production of drawings and their submission to the assembly and production department.

The purchasing and production phases run in parallel. The production department works on parts that do not need to be purchased but can be manufactured, thus saving the budget and eliminating the lead time that would have been incurred by purchasing these parts. In the purchasing phase, materials and parts are ordered according to the schedule. The purchasing department is responsible for delivering the material according to the project schedule. If some items, such as industrial robots, have longer lead times, they can be ordered during the design phase.

Once the items are received from the production and purchasing departments, the assembly phase begins. In this phase, all the delivered items are assembled into a single unit according to the relevant documentation. The assembly phase also includes debugging. After debugging and assembly inspection, the production and assembly line is ready for export. However, no production line can be delivered to the customer without a FAT

(Factory Acceptance Test). This test is a comprehensive functionality check and includes software debugging of the line.

If the FAT test is successful, there is nothing stopping the production and assembly line from transporting it to the customer. Transport can be internal or external. For external transport, information about the load, such as weight and maximum dimensions, is required. Once the production and assembly line has been transported to the customer, external assembly takes place.

External assembly takes place at the customer's site. Once all parts have been assembled, the machine is tested for safety on site. Documents are then handed over to the customer.

The final handover of the production and assembly line to the customer is followed by a SAT test (Site Acceptance Test). After approval, all acceptance documents are signed and the handover process is complete.

At the end of the project, there is one final point, the project closure, where the whole design and production process is evaluated retrospectively and lessons learned are written down for future projects.

With the definitive closure of the project and the accompanying documentation, the production line is ready for management, which must comply with the company's warranty conditions. Service and warranty repairs are then handled and covered by the service department.

3. Compressed Air as a Working Medium on Production and Assembly Lines

Compressed air is used extensively in all areas of industry, commerce and everyday life. The possible applications are varied and extensive. Methods of technical use:

- Compressed air transport in mechanisation and automation
- Clamping with compressed air
- Compressed air spraying
- Pneumatic drive systems
- Compressed air blowing
- Compressed air testing and inspection
- Using compressed air for process control

The use of compressed air as a medium has its advantages but also disadvantages. Proper treatment of compressed air will result in the following benefits:

- Increased durability of consumer equipment.
- Consistent product quality.
- Pneumatic piping without condensation and rust.
- Reduced breakdowns.
- Reduced service costs.
- Reduced pressure loss due to leakage and flow resistance.
- Reduced energy consumption due to reduced pressure loss [8, 9].

However, if a situation arises where dirt and water from the atmospheric air remain in the compressed air, the consequences can be unpleasant. This applies to pipework and consumer equipment, but manufactured products can also be adversely affected if the quality of the compressed air is poor. In some applications, the use of compressed air without proper treatment is dangerous and poses a health risk.

3.1 Air, Air Leaks and Air Monitoring

Compressed air loss is the consumption of air (leakage) in the piping without work being done. These losses can reach 25% of the volume losses of the compressor under unfavourable conditions. The causes are many:

- Leaking valves
- Leaking bolted and flanged joints
- Leaking welding or brazing joints
- Damaged hoses and hose connections
- Defective solenoid valves
- Stuck float drains
- Improperly installed dryers, filters and service equipment
- Loose couplings

Leaks in the pipework act as nozzles from which compressed air escapes at high velocity. These leaks consume compressed air 24 hours a day. The energy required to compensate for this loss can be significant. It does not cause physical injury, but the resulting costs can seriously reduce the cost-effectiveness of a pneumatic system.

In addition to being a source of wasted energy, leaks can contribute to other operational losses such as:

- **Loss of productivity:** If tools are not getting the right amount of air, they will not work efficiently. Employees may take longer to complete a task due to a lack of power, or they may struggle to get the results they need.
- **Increased downtime:** With air compressor leaks comes air compressor repair. The more the system is down, the less work can be done. Maintenance replaces production time.
- **Increased capacity:** If more air is needed than the leaky system can handle, more capacity must be added to compensate for the leak. This addition increases the electrical charge and can wear out the system more quickly [10].

3.2 Identifications of Air Leaks in the Industry

When an air leak is suspected in production and assembly systems, the leak identification phase can be initiated. There are a number of ways to identify leaks and the choice of the appropriate method is influenced by a variety of factors.

Determining leakage by emptying the vessel - this method of measurement is only suitable for compressed air systems where the pipe network is less than 10% of the volume of the vessel. Otherwise, the inaccuracy of the measurement will be too high. The following formula is used to roughly quantify the leakage volume.

Determining leakage by measuring compressor duty cycle - appliances on the network are shut down. Due to a leak in the system, compressed air is consumed and the pressure in the network

drops. This amount of leakage must be replaced by the compressor. The total running time of the compressor is measured over a period of time. To obtain a meaningful result, the measurement period should include at least 5 compressor switching intervals.

Hissing Air Detection by Sound - Leaking air can be heard and felt by touch [11].

Soapy water detection - A method using a spray bottle filled with mild soapy water. It is a cheap and easy way to find leaks. Simply spray the system connections with soapy water [12].

Ultrasonic method - Specialised equipment and a qualified technician should be used to detect compressed air leaks reliably and as quickly as possible, especially in noisy environments. Ultrasonic leak detection equipment is the best tool for identifying leaks. Its regular use can help maintain a consistent and high level of compressed air system functionality. Regular leak checks reduce wasted energy, unnecessary wear and tear on air compressors and help prevent air compressor downtime. The form of ultrasonic technology used to detect compressed air leaks is in-air ultrasonics. Air ultrasonic is defined as high-frequency sound waves that are above the range of normal human hearing. Most ultrasonic equipment is capable of receiving or detecting high-frequency ultrasonic sound waves in the frequency range of 20 kHz to 100 kHz. For ultrasonic leak detection, an ultrasonic device with frequency tuning capability is recommended and the suggested frequency setting is 40 kHz. In compressed air and gas leak detection, the source of ultrasound is turbulence. When compressed air flows through a pipe, it moves in a laminar flow. This type of flow means that all particles flow in the same direction and in parallel. If there is a leak in the pipe, the air flow around the leak changes to turbulent flow. The air does not flow in the same direction. This turbulent air creates noise which is interpreted by ultrasonic detectors. The detector works in one of two ways. Either it is connected to headphones and produces a sound that gets louder when there is a leak, or it has a screen that displays the ultrasonic waves. The wave will change significantly as the sound gets louder. A representation of turbulent and laminar flow can be seen in Figure 2 [13, 14].

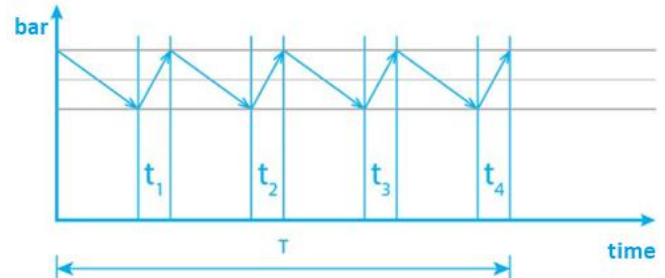


Fig. 2 Graph of turbulent and laminar flow representation

Other sources of high-frequency noise include ionisation, friction and impact. Ionisation occurs in electrical components when the air around an electrical connection becomes a conductor, producing a constant 'hum'. A by-product of the ionisation process is nitric acid. Therefore, if ionisation is allowed to continue unchecked, the nitric acid will corrode and degrade the connections, leading to more severe failure conditions.

A particular type of device used in the industry is called the SONAPHONE Pocket from Sonotec, shown in Figure 3.



Fig. 3 SONAPHONE Pocket from Sonotec

3.3 Measures to Minimise Compressed Air Losses

Refurbishment of the compressed air network - if the amount of leakage found in the compressed air network is significantly higher, refurbishment of the network should be considered. When refurbishing the pneumatic system, measures should be taken to reduce compressed air losses:

- Replace leaking valves, leaking hoses and hose couplings
- Weld leaking pipework
- Improve compressed air treatment
- Remove harmful contaminants such as water, oil and dust from the compressed air
- Inspect solenoid valves [15]

A common source of compressed air loss that is not technically a leak is improper use. When employees use air for a purpose for which it is not intended, such as a blower to clean or cool electrical panels or cabinets, they create another source of energy loss. Alternative uses must be identified and workers should be allowed to use more efficient and appropriate tools for the job. These tools may include utility nozzles designed specifically for the task or cooling units designed for electrical panels or cabinets. This will allow employees to use less energy and can help take some of the load off the air compressor system.

Another measure to reduce compressed air consumption is the design of leak detection software. The leak detection software includes introduction of leak prevention checks using mobile software:

- A software/database to flag regular leaks
- A software/database to locate, identify and repair leaks

The FESS portal is such software. The FESS Portal is an online tool for documenting and managing energy savings data and its benefits are:

- **Immediate access to results** - no need to wait for a service report, all activities can be documented immediately.
- **Worldwide availability of results** - an advantage for international companies where one report is available to all authorized staff.
- **Ideal for recurring services** - benchmarking results and transparent documentation of savings.

There are several key elements required for a successful leakage elimination software, which are listed below:

- Compressed air consumption baseline data

- Assessment of leakage losses
- Air leak costing
- Identify leaks
- Document the leaks
- Prioritise repair of leaks
- Document repairs
- Compare baselines and publish results [16]

A proper and functioning leak repair program for compressed air systems is critical to maintaining the efficiency, reliability, stability and cost-effectiveness of any compressed air system. System identification and logging of fault/leak information provide information on the nature and cause of leaks and the repairs required, helping users to establish a systematic and sustainable leak management program.

3.4 Identification of Air Leaks in Relation to the Available Pneumatic Control Scheme

Identification begins with a description of all the pneumatically actuated components, their different types, the operating principle and examples of the use of these basic automation elements. The identification of the main reference objects, which are most often identified as the cause of air leakage, is included in the design part of the universal air leakage measurement principle. Based on the maximum allowable leakage value $QL_{max}=10 \text{ ml.min}^{-1}$, the pressure drop identification and flow measurement methods shall be specified.

The input data is the basis for selecting the appropriate flow measurement method using a flow meter. There are a large number of flowmeters on the market that operate on different principles. One of these, for example, is the thermal mass flowmeter, which is the most resistant to unforeseen pressure changes in the measuring system. It can provide accurate measurements of leakage air flow [17].

The pneumatic circuit shown in Figure 4 is a universal connection between the logic link compression device and the pneumatic circuit, which can be used to test any pneumatic component. It is necessary to determine the appropriate locations where it would be appropriate to use mechanical regulators or regulators with adjustable output pressure.

4. Conclusion

Air leakage testing is an essential part of the manufacturing and assembly process of pneumatic components by which any manufacturer will want to ensure the required quality and proper functioning of the product. Identifying air leaks is important for the proper functioning of the system and is also essential for saving financial costs for the company. Some of the basic measures that could be taken are:

- Reducing inappropriate use of compressed air
- Reducing the pressure in the compressors, reducing the temperature of the intake air
- Ensure adequate storage capacity
- Recovering waste heat from the air compressor
- Reducing leakage

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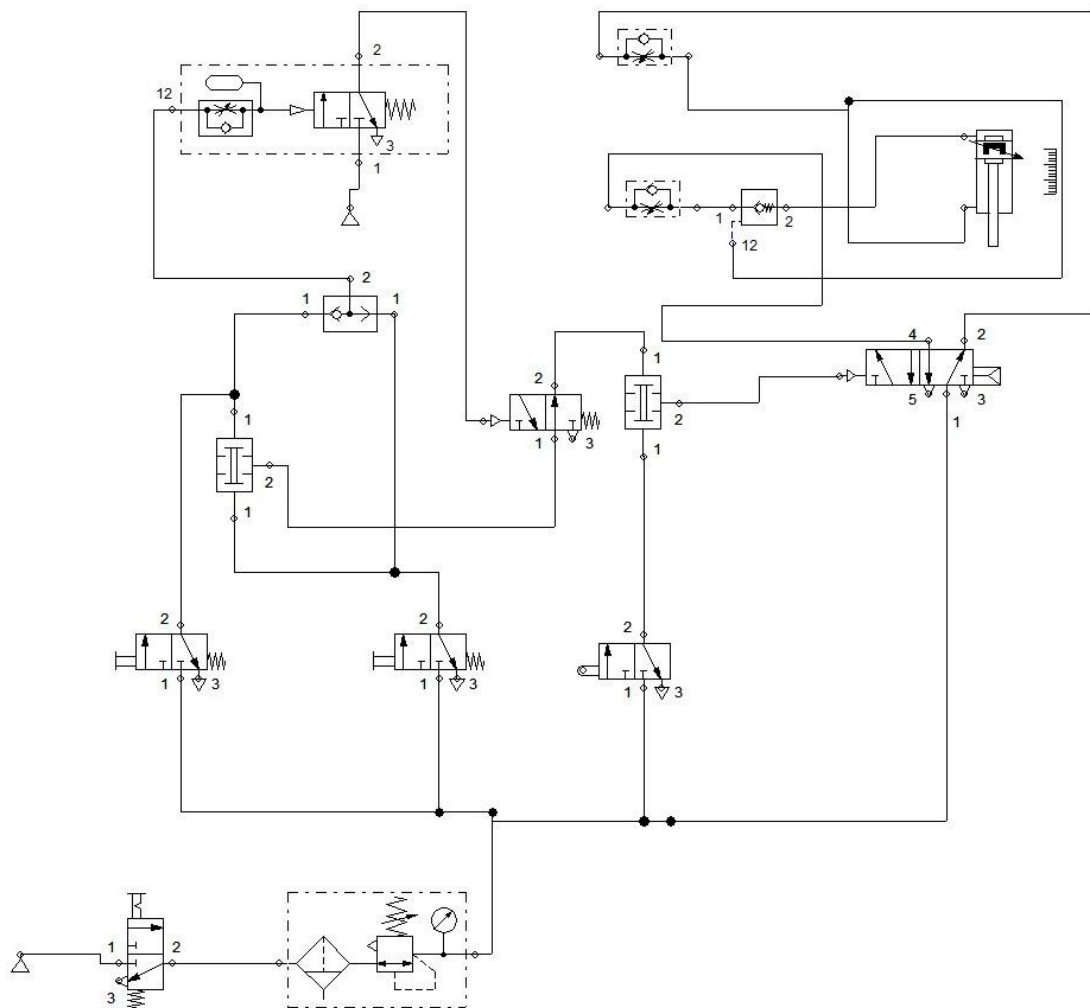


Fig. 4 *Pneumatic circuit of pressing process*

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The influence of electric discharge and solutions of surfactants on the filtration characteristics of porous, liquid-saturated media

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Abstract: The results of studies of the behavior of rocks with fluids under impulse pressure, which is induced by an electric discharge in solutions of surface-active substances (surfactants), are given. The mechanism of influence of high-voltage electric discharges and mechanical waves induced by them on the behavior of combined objects (porous rocks, hydrocarbon fluids filling their pores – oil and hydrogen surfactant solution, solid mineral sediment) is revealed.

It was found out that the use of a water-oil emulsion as a working medium for high-voltage electric discharge significantly (2 times for sandstones and 1.5 times for carbonates) increases the efficiency of the electric discharge effect compared to water.

The complex action of the electric discharge and the aqueous 0.3% solution of NMK-21, used as a working medium, leads to a synergistic effect in increasing the permeability of rocks by twice the sum of the effects for each component.

Work results can be used in the development of scientific foundations of managing the filtration properties of porous materials of various origins.

KEYWORDS: HIGH VOLTAGE ELECTRIC DISCHARGE, ROCKS, OIL, POROUS MEDIUM, SANDSTONE AND CARBONATE CORES, SURFACTANT SOLUTION.

1. Introduction

The structure of porous materials of natural and man-made origin changes when liquids are filtered through them due to the contamination of the pores by various types of sediments, both brittle and viscoplastic. Such phenomena are characteristic to most modern technologies: the fine purification of liquid and gaseous media, the movement of hydrocarbon fluids in capillary-porous media, their transportation and initial preparation during oil processing in the oil industry, etc. It is known that the most effective influence on the structure of porous materials can be achieved by using physical methods of impact (electromagnetic, vibration, ultrasonic, etc.). Each of these methods has many advantages, but they have one common drawback – none of them allows controlling the process of regulating the structure (and thus the properties) of porous materials and the dynamics of liquid filtration in them [1].

High voltage electric discharge (HVED) in liquid is an efficient physical method of impacting porous materials, which gives the way to control the characteristics of treated material by varying treatment parameters [1]. HVED can be realized in the solutions of surfactants, which increases treatment efficiency.

Therefore, the **goal of the work** is to study the impact of high voltage electric discharge parameters and the presence of surfactants solutions on the filtration characteristics of porous media, saturated with liquid.

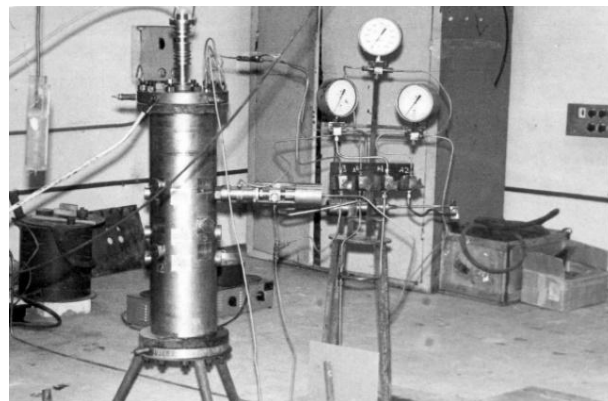
2. Methodology

Filtration properties of porous, liquid-saturated materials, namely sedimentary rocks that form reservoirs of hydrocarbon fluids, are characterized by such basic indicators as porosity and permeability [2]. It is known that about 60% of the world's oil reserves are confined to sandy layers and sandstones, 39% – to carbonate deposits, and 1% – to weathered metamorphic and igneous rocks. Therefore, studies of the dynamics of changes in filtration characteristics with the simultaneous application of high-voltage electric discharge in various solutions of surfactants were performed on sandstone and carbonate cores.

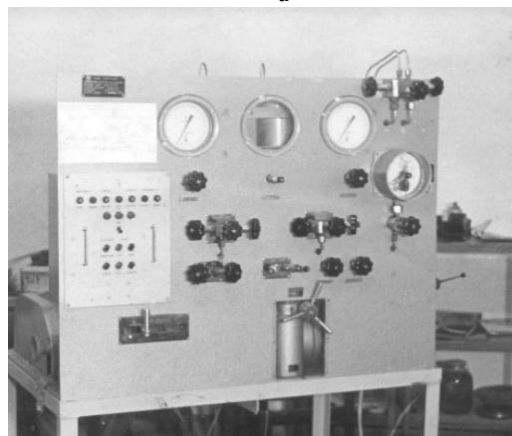
The study of the dynamics of changes in the permeability of the cores as a result of the impact of a high-voltage electric discharge on them in various surfactant solutions was carried out on the high-pressure experimental stand (see Fig. 1). The high-pressure chamber, in which the core holder is built perpendicular to the axis (see Fig. 1, a), is connected to the stand for studying the permeability of cores, which is part of the "AKM-collector" equipment set (see Fig. 1, b).

The study of the physical features and principles of control of the change in the filtration characteristics of porous, liquid-saturated

materials during an electric discharge in aqueous electrolytes (water), aqueous solutions of surfactants, water-oil emulsions (WOE) with water content of 30% and WOE with the addition of surfactants was performed on sandstone and carbonate cores contaminated with asphalt-resin-paraffin deposits (ARPD).



a



b

Fig. 1. The stand for researching the filtration characteristics of reservoir rocks: a – high-pressure chamber with a core holder; b – "AKM-collector" equipment set.

The research results presented in the work [3] showed that NMK-21, the mixture of anionic and nonionic surfactants, was the best of the investigated surfactants in terms of all basic properties, therefore, 0.3% aqueous solutions and additives in WOE were prepared on the basis of this surfactant.

3. Results and discussion

Fig. 2 shows the dynamics of changes in the permeability of sandstones and carbonates during filtration of an aqueous 0.3% solution of NMK-21, which is expressed as k_1/k (k – permeability of the contaminated core; k_1 – permeability of the core washed with a surfactant solution) Stabilization of rock permeability occurs after approximately 30 minutes (for sandstones) and after 40 minutes (for carbonates) of filtration. It should be noted that filtering water and WOE through a contaminated ARPD core does not lead to an increase in permeability, because washing does not occur.

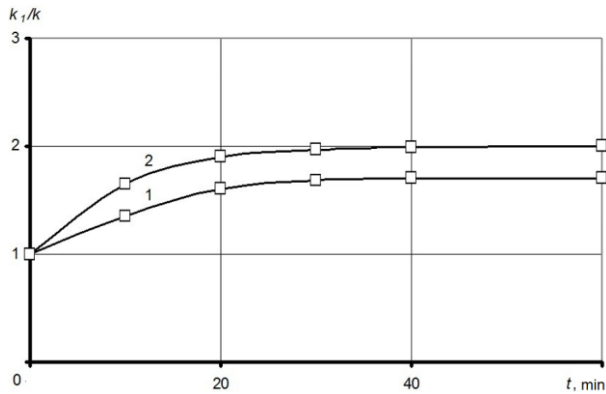


Fig. 2. The dynamics of changes in rock permeability under the impact of 0.3% NMK-21 solution: 1 – carbonate; 2 – sandstone.

Changes in permeability k_n/k (k_n – permeability after electric discharge treatment) of sandstones and carbonates that occur as a result of high-voltage electric discharge treatment in water and WOE are presented in Fig. 3. It is obvious that the use of WOE as a working medium for HVED allows significantly (2 times for sandstones and 1.5 times – for carbonates) increasing the efficiency of the electric discharge treatment compared to using water. The increase in the permeability using the high-voltage electric discharge in water, and in WOE for carbonates, was slightly (~35%), higher than the effect achieved by using the surfactant solution.

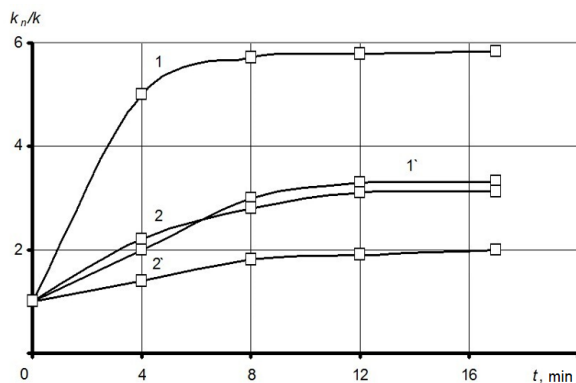


Fig. 3. The dynamics of changes in the permeability of rocks after HVED treatment: 1, 2 – sandstones; 1', 2' – carbonates; 1, 1' – water-oil emulsion; 2, 2' – water.

The combined action of the electric discharge and the aqueous 0.3% solution of NMK-21, used as a working medium, leads to a synergistic effect in increasing the permeability of rocks by twice the sum of the effects for each component separately (see Fig. 4).

In a theoretical study [2] it was shown that the initial stage of pulsed loading of a porous medium is characterized by a close to linear relationship between the number of sequentially performed electric discharges and the mass of deposits removed from the pores of the porous medium (which leads to an increase in porosity). The change (increase) in the permeability of porous rocks depends on

the change (increase) in their porosity in the degree of ~10. Accordingly, the change in the permeability of a simple material within the limits of its saturation with a surfactant solution is proportional to the number of consecutive electric discharges taken in the 10th degree.

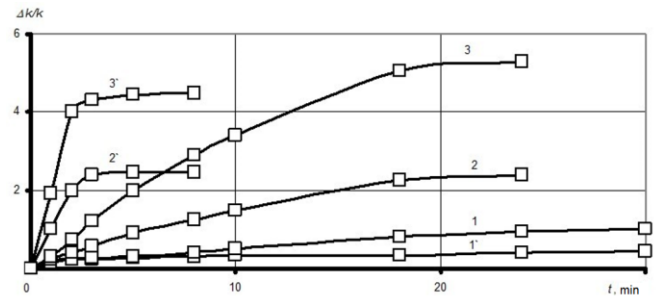


Fig. 4. Change in permeability of sandstones (1, 2, 3) and carbonates (1', 2', 3') after the treatment with: 1, 1' – 0.3% aqueous solution of NMK-21; 2, 2' – electric discharge; 3, 3' – electric discharge + 0.3% aqueous solution of NMK-21.

Thus, despite the relatively small amount of sediment removed from the pores in one discharge, a sequence of discharges can lead to a significant increase in the permeability of the porous material.

The results of theoretical studies are confirmed by the experimental data that the usage of a water-oil emulsion with an addition of surfactant as a working medium for HVED treatment leads to a significant increase in the porosity of sandstones (up to 3 times) and carbonates (up to 2.5 times) [4]. Such a change in porosity may indicate that when a porous medium is unevenly loaded with a surfactant solution, its structure is restructured due to artificially created porosity (microstructural studies were performed on a Neofot-32 optical microscope). As the influence of rock porosity on its permeability is significant, the permeability coefficient of the rock does not increase linearly with the increase in porosity. Reconstruction of the structure due to additional porosity as a result of the saturation of the rock with liquid has the greatest influence on the dilatational densification of the rock [5].

Microstructure studies of cores using an optical microscope also indicate structural changes in the rock due to an increase in pore size (see Fig. 5).

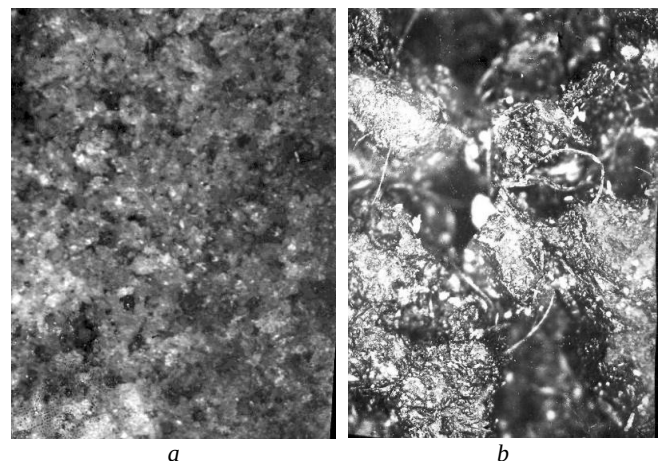


Fig. 5. The microstructure of sandstones ($\times 50$; optical microscope): a – before electric discharge influence; b – after electric discharge influence.

It is known that in natural materials with different pore sizes, the porosity depends on the size distribution of the pores. Therefore, studies of the size distribution of pores before and after electric discharge exposure were performed (see Fig. 6 and Fig. 7). For this, the entire data array was divided into mass groups, the interval of values in the group was determined arbitrarily, according to the obtained minimum and maximum values

For sandstones before exposure to HVED the maximum pore size was 12 μm (see Fig. 6, curve 1), and as a result of electric discharge treatment in WOE environment with the addition of 0.3% NMK-21, the pore size increased from 80 to 560 μm , and most pores have a size of 300 μm (see Fig. 7, curve 2). A similar picture is also observed for carbonates, and if before exposure the maximum pore size of carbonates was 2 times smaller (see Fig. 6) than that of sandstones, then after exposure the spread of pore size values became practically comparable (see Fig. 7, curves 2, 4).

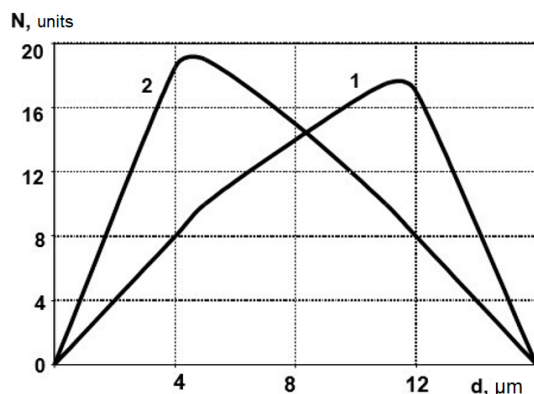


Fig. 6. The distribution of pores in rocks by size before electric discharge treatment: 1 – sandstones; 2 – carbonates.

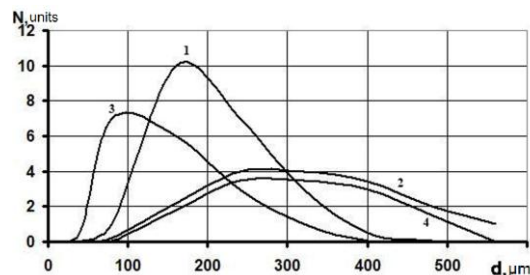


Fig. 7. The distribution of pores in rocks by size after electric discharge treatment: 1 – sandstones (water-oil emulsion); 2 – sandstones (water-oil emulsion + NMK-21); 3 – carbonates (water-oil emulsion); 4 – carbonates (water-oil emulsion + NMK-21).

The electric discharge action in WOE also led to an increase in the pore size from 20 to 350 μm in carbonates, with most pores 80 μm in size, as well as from 40 to 400 μm in sandstones with most pores 180 μm in size.

Thus, when the porous medium is unevenly loaded with a surfactant solution in the WOE, which is used as a working medium during an electric discharge, its structure is rearranged due to artificially created porosity. Such loosening of porous materials is called dilatancy [5].

By comparing the results of the research, it is possible to substantiate the factors that contribute to the manifestation of a synergistic effect in changing the filtration characteristics of porous materials (rocks saturated with hydrocarbon liquids) under the complex influence of a high-voltage electric discharge and a solution of surfactants. These factors should include the activation of the surfactant solution during high-voltage electric discharges due to gas formation in the medium and its foaming during intensive mixing of the gas-liquid medium in the presence of a foaming agent (nonionic surfactant components). The electric discharge treatment of a 0.3% solution of NMK-21 allowed to achieve almost complete (93%) removal of ARPD, which is confirmed by the results of restoring the porosity of the cores to 90%. ARPD removal is enhanced as a result of better adsorption of surfactants on the solid surface, which is facilitated by foam bubbles. This fact is also confirmed by the increase in the effect of changing the permeability of rocks when adding 0.3% of the NMK-21 solution to WOE. In the WOE environment, gas formation begins almost from the first pulses due to the decomposition of the

medium by the plasma channel, so the foam bubbles immediately enter the pores of the rock together with the filtration flow and are adsorbed on the surface of the pores covered with ARPD.

Taking into account the fact that the value of the ionization potential u_i of carbon atoms $u_i(\text{C}) \approx 11.3$ eV is lower compared to oxygen and hydrogen atoms: $u_i(\text{H}) \approx u_i(\text{O}) \approx 13.6$ eV, as well as the lower average breaking energy of C–H, C–C bonds (WC–C–H ≈ 90 kcal/mol, WO–H ≈ 100 kcal/mol) [6], the percentage ratio of the synthesized gas phase due to contact with plasma of the discharge channel during HVED in a hydrocarbon medium is higher than in water. The fact that the rate of change in the permeability of the cores treated in the environment of WOE + NMK-21 is higher than in case of treatment in aqueous solutions of NMK-21 is probably also related to this.

The next most important factor that leads to a synergistic increase in the filtration characteristics of porous materials is the pulse reciprocating nature of the activated surfactant impact. The pressure wave at the compression stage directs the surfactant solution into the porous medium, and at the decompression stage this liquid moves in the opposite direction. Such repeated reciprocating movement of the solution stimulates the washing away of deposits, which contributes to the creation of a synergistic effect in saturated porous media.

The reduction of surface tension at the interface of the media during the electric discharge treatment of surfactant solutions is important in creating a synergistic effect. As a result of the decrease in surface tension at the interface of the media, the hydrophilicity of the rock grains and the permeable filtering capacity of the surfactant solution in the densest materials increases.

Previously, in works [7–12], a significant dependence of the change in permeability on the amplitude of the pressure pulse in the medium of aqueous electrolytes and in the WOE was noted, which was the reason for considering compression waves as the main mechanism of influence on porous saturated mediums. Currently, guided by these works, it should be clarified that compression waves destroy fragile sediments in the zone of perforation holes, then, repeatedly reflected and transformed into stress-tensile waves, they develop new fracture channels. Drilling fluid penetrating at high speed into the perforation holes and then into the rock also contributes to the destruction of sediments and the development of cracks. At the same time, pressure fluctuations characteristic to pulsed electric discharge impact, while pressure changes alternately in magnitude and direction, contribute to the movement of liquid in the pores of the rock, thereby intensifying the filtration processes.

At high hydrostatic pressures, characteristic to deep layers of porous materials saturated with hydrocarbon liquids, the composition of the liquid in which the high-voltage electric discharge is carried out is one of the main factors that determines the effectiveness of the impact on the structure of porous materials and the dynamics of filtration of hydrocarbon fluids in them. Therefore, the use of a medium, that is chemically active with the type of deposits in porous media, as a working fluid for HVED treatment will contribute to a significant expansion of the possibilities of using electric discharge technology for the intensification of filtration processes in porous materials.

4. Conclusions

On the basis of the conducted research, it is possible to formulate the main factors that contribute to the creation of a synergistic effect in changing the filtration characteristics of porous materials (rocks) during the electric discharge treatment in surfactant solutions [12, 13]:

- activation of surfactant solutions, which occurs as a result of gas formation and foaming (if it contains a foaming agent) during high-voltage electric discharges;
- alternating pressure (compression-decompression) in surfactant solutions filtered into the porous medium, which stimulates the effect of washing pores from deposits and contributes to the increase of the area covered by the influence;

- adsorption of the activated surfactant on the rock, which helps to reduce the surface tension at the interface between the liquid and solid phases (the walls of the filtration channels) and increases the permeable filtration capacity of the surfactant solution;
- adsorption of activated surfactant on rock washed from ARPD, which prevents the further growth of ARPD crystals and their adhesion to the surface, contributing to an increase in the time of the treatment effect;
- adsorption of activated surfactant on ARPD crystals in the oil located in the pores, which leads to the destruction of its structural framework, improving its mobility during filtration (the ultimate shear stress, shear stress and dynamic viscosity decrease);
- adsorption of activated surfactant in pores and microcracks, which contributes to the creation of a wedging effect due to pulse saturation with the filtered medium and dilatational densification of the rock due to the restructuring of its structure with the formation of additional porosity.

Acknowledgements

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Manufacturing Principals of Metal processing Lasers

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Abstract: This paper provides a concise overview of the manufacturing principles of metal processing lasers. Metal processing lasers are advanced tools used in various manufacturing industries for cutting, welding, and shaping metal materials. The presented highlights include the fundamental principles governing laser-based metal processing techniques, including laser beam generation, focusing, and interaction with metal surfaces. It also emphasizes the importance of precise control parameters, such as power, pulse duration, and wavelength, to achieve optimal processing results. Understanding these principles is crucial for enhancing productivity, quality, and efficiency in metal manufacturing processes.

1. Introduction to Laser characteristics

Lasers, which stands for "Light Amplification by Stimulated Emission of Radiation," operate based on the principles of quantum mechanics and electromagnetic radiation. To understand the operational principles of lasers more scientifically, we need to explore the concepts of gain medium, pumping mechanisms, optical resonators, and the underlying mathematical formulations involved.

The fundamental component of a laser is the gain medium, which is typically composed of solid-state, gas, or semiconductor materials. The behavior of the gain medium can be described using quantum mechanical concepts. In a simplified model, the gain medium consists of many identical atoms or molecules, each possessing discrete energy levels. The energy levels are typically represented by quantum numbers, and the energy difference between the levels can be calculated using the Planck's equation:

$$E = hf.$$

Where E is the energy difference, h is Planck's constant, and f is the frequency of the emitted photon. When the gain medium is excited, either through electrical discharges, optical pumping, or electrical current injection, the atoms, or molecules within it undergo transitions between energy levels. These transitions can be quantified using Einstein's coefficients, which describe the probability of different processes: absorption, spontaneous emission, and stimulated emission. The rate of stimulated emission, which contributes to the amplification of light in the gain medium, can be expressed mathematically using the following equation:

$$R_{stim} = B21\rho(E)\varphi(E),$$

Where R_{stim} is the rate of stimulated emission, $B21$ is the Einstein coefficient for stimulated emission, $\rho(E)$ is the population density of excited states, and $\varphi(E)$ is the spectral distribution of photons.

The pumping mechanism provides the necessary energy to excite the gain medium. In gas lasers, the excitation is achieved through electrical discharges. The discharge current, voltage, and gas pressure can be controlled to optimize the energy transfer to the gas atoms or molecules. In solid-state lasers, optical pumping is employed, where an external light source illuminates the gain medium, transferring energy to the atoms or molecules. The rate of energy transfer can be calculated using the concept of cross-section, which quantifies the probability of interaction between photons and the gain medium. For example, in solid-state lasers, the rate of energy transfer through optical pumping can be described using the following equation:

$$dN/dt = \eta \cdot \sigma_{pump} \cdot \varphi_{pump} - (\sigma_{em} + \sigma_{abs})N,$$

where dN/dt is the rate of change of excited state population, η is the quantum efficiency, σ_{pump} is the absorption cross-section, φ_{pump} is the spectral distribution of the pumping light, σ_{em} is the emission cross-section, σ_{abs} is the absorption cross-section, and N is the number of excited atoms or molecules.

2. Optical resonator

The optical resonator Fig. 1, consisting of two mirrors, plays a crucial role in laser operation. The behavior of the resonator can be analyzed using the principles of wave optics. The reflections from the mirrors create standing waves within the resonator, and the constructive interference of these waves enhances the intensity of the laser beam. The resonant condition for constructive interference can be described mathematically using the equation:

$$2L = m\lambda,$$

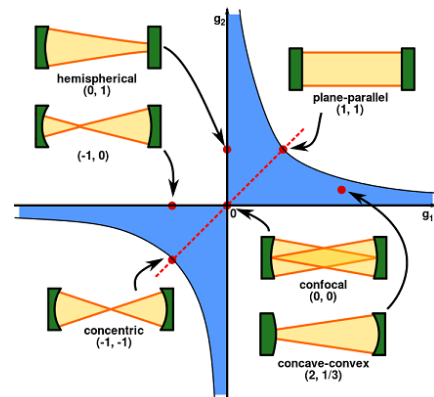


Fig. 1 Laser resonator stability

Where L is the distance between the mirrors, m is an integer representing the mode number, and λ is the wavelength of the laser light. Furthermore, the extraction of laser light from the resonator can be understood using the concept of transmittance [8]. The transmittance of the partially reflective mirror determines the fraction of light that exits the resonator and becomes the laser output.

3. Einstein coefficients, cross-sections, and population densities

In addition to the concepts discussed earlier, the behavior of lasers can be further analyzed using rate equations, which describe the time evolution of the populations of different energy levels within the gain medium. These rate equations consider the rates of absorption, spontaneous emission, and stimulated emission processes. For a simple two-level system, where there are only two relevant energy levels in the gain medium, the rate equations can be written as follows:

$$dN1/dt = - (B21\rho\phi + \sigma_{abs}\phi)N1 + B12\rho\phi N2 + R_{sp},$$

$$dN2/dt = B21\rho\phi N1 - (B12\rho\phi + \sigma_{em}\phi)N2,$$

$N1$ represents the population of the lower energy level, $N2$ represents the population of the upper energy level, $B21$ is the Einstein coefficient for stimulated emission, $B12$ is the Einstein coefficient for stimulated absorption, ρ is the population density, ϕ is the spectral distribution of photons, σ_{abs} is the absorption cross-section, σ_{em} is the emission cross-section, and R_{sp} is the rate of spontaneous emission.

These rate equations describe the competition between absorption and stimulated emission processes, as well as the interaction between the upper and lower energy levels. The rates of these processes depend on the population densities, spectral distribution of photons, and the respective Einstein coefficients and cross-sections. To achieve laser operation, a condition called population inversion must be established, where the population of the upper energy level is greater than that of the lower energy level. This condition ensures that stimulated emission dominates over absorption, leading to the amplification of light.

The population inversion can be quantitatively described by the following equation:

$$\rho = (N2 - N1) / (N2 + N1),$$

This equation represents the fractional difference between the populations of the upper and lower energy levels.

4. Threshold condition

Another important parameter in laser operation is the threshold condition Fig. 2, which determines the minimum amount of pump energy required for laser action. The threshold condition can be expressed in terms of the gain and losses within the laser system. The gain refers to the net amplification of light, while the losses account for factors such as mirror reflectivity, absorption, and scattering. Mathematically, the threshold condition can be written as:

$$G = \exp[(\sigma_{em} - \sigma_{abs})L] - Rl,$$

where G represents the net gain, σ_{em} is the emission cross-section, σ_{abs} is the absorption cross-section, L is the length of the gain medium, and Rl represents the round-trip losses due to mirror reflectivity, absorption, and scattering. When the pump energy exceeds the threshold condition, the laser system reaches a point where the net gain compensates for the losses, and laser action is sustained [7].

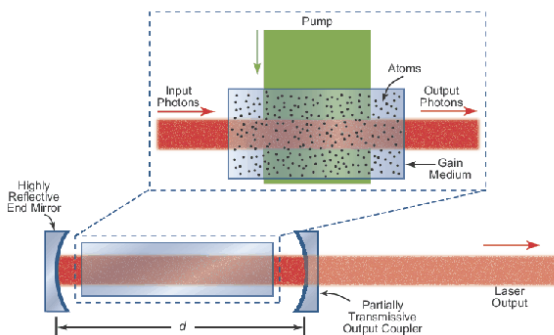


Fig. 2 Threshold condition

Moreover, the output characteristics of lasers, such as the beam profile and spectral properties, can be analyzed using mathematical techniques such as Fourier analysis, Gaussian beam propagation, and spectral analysis. These mathematical tools provide insights into the spatial and spectral distribution of the laser beam, enabling the optimization of laser performance for specific applications.

5. Coherence of emitted light

One more crucial aspect of laser operation is the coherence of the emitted light. Coherence refers to the correlation between the phases of different waves in the laser beam. In lasers, this coherence arises from the stimulated emission process, which ensures that the emitted photons have the same frequency and phase as the incident photons. The coherence length, often denoted as L_c , represents the distance over which the laser beam maintains its coherence. It can be calculated using the formula:

$$L_c = \lambda^2 / \Delta\lambda,$$

Here, λ represents the wavelength of the laser light, and $\Delta\lambda$ represents the spectral linewidth, which is a measure of the range of wavelengths present in the laser output. The coherence length determines the spatial and temporal characteristics of the laser beam. A longer coherence length indicates that the laser beam can travel a greater distance without significant changes in its phase relationship. This property is crucial for applications such as holography, interferometry, and long-distance communication.

In addition to coherence, the spectral characteristics of laser light are also of great importance. The spectral linewidth, mentioned earlier, represents the range of wavelengths present in the laser output. In an ideal laser, the linewidth would be infinitesimally narrow, consisting of a single precise wavelength. However, in practical lasers, there are various factors that contribute to linewidth broadening.

One of the main factors is known as spontaneous emission. While stimulated emission is the desired process in laser operation, spontaneous emission occurs randomly and is not phase-correlated with the incident photons. As a result, it leads to broadening of the spectral linewidth. The linewidth broadening due to spontaneous emission can be quantified using the Schawlow-Townes formula:

$$\Delta\nu \approx (2\pi h\nu^2 / P) / (2\tau),$$

Here, $\Delta\nu$ represents the spectral linewidth, h is Planck's constant, ν is the frequency of the laser light, P is the power output of the laser, and τ is the lifetime of the excited state in the gain medium. To achieve a narrow spectral linewidth, lasers often employ techniques such as cavity stabilization, which involves precise control of the resonator length and temperature. These techniques help minimize the effects of factors contributing to linewidth broadening, resulting in lasers with narrower spectral profiles.

6. Spatial distribution

Another significant characteristic of laser light is its spatial distribution, often described using parameters such as beam divergence and beam quality. The beam divergence represents the spreading of the laser beam as it propagates in free space. It is influenced by factors such as the size and shape of the gain medium and the properties of the optical resonator.

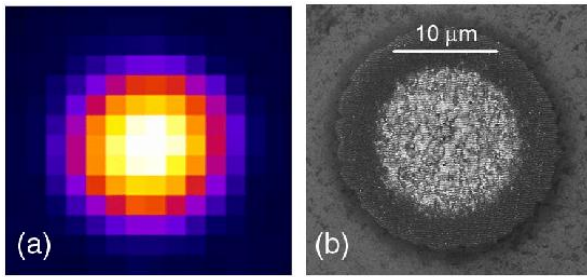


Fig. 3
(a) Spatial distribution of laser beam intensity in the irradiation spot (248 nm, 450 fs) and (b) SEM image of the pattern. The pixel size is 5 μm (a).

The beam quality, often quantified using a parameter called the M^2 factor, provides information about the spatial profile of the laser beam. A lower M^2 factor indicates a more focused, diffraction-limited beam, while a higher M^2 factor suggests a broader, less focused beam [6]. The beam quality is determined by factors such as the design of the optical resonator, the mode structure within the resonator, and the quality of the gain medium. Achieving a high beam quality is crucial for applications such as laser material processing and high-precision measurements. To analyze and optimize the spatial distribution of laser light, mathematical techniques such as Fourier optics and beam propagation methods are employed. These methods consider diffraction effects and the interplay between wavefronts and optical components to model and predict the behavior of laser beams.

7. Conclusion

Lasers operate based on a combination of quantum mechanics, wave optics, and statistical physics. The behavior of lasers can be analyzed using rate equations, population inversion calculations, and threshold conditions. They exhibit important characteristics such as coherence, spectral properties, and spatial distribution. The coherence length describes the distance over which the laser beam maintains its phase relationship, while the spectral linewidth characterizes the range of wavelengths present in the laser output. The spatial distribution of the laser beam is described by parameters like beam divergence and beam quality, which impact applications requiring precise focusing or broad coverage. Mathematical tools and analysis techniques enable scientists and engineers to understand and optimize these characteristics, leading to improved laser performance for a wide range of applications.

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Determination of the quality of welded joints after repairs of hot water pipes

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Abstract: The paper presents the results of an investigation aimed at analysing the current state of the art in the construction of hot water piping systems. Weld joint analysis and evaluation is an essential part of the inspection of hot water pipelines to ensure their safe operation. The practical part of this work is focused on the analysis and evaluation of the weld joint of original and new hot water piping. Destructive and non-destructive weld joint testing methods were applied to carry out the evaluation and analysis, the results of which were used to compare the welded joints on the hot water pipeline.

Keywords: WELDING, PIPELINE, TESTING, QUALITY

1. Introduction

The renewal of piping systems is a hot topic all the time. The service life of piping systems is limited by many degradation factors. Depending on the purpose of the piping system, primary and secondary degradation factors can be classified [1-3]. Drinking water piping systems, sewage systems as well as hot water piping systems are primarily exposed to adverse corrosion conditions and consequently, e.g., to pressure or stresses due to dilatations caused by temperature changes of the flowing medium, or to the effects of external weather conditions [4-8]. The lifetime of piping systems is limited to several decades and extensive replacement of these systems is currently being carried out due to accumulating breakdowns. The mass construction of housing estates and urban districts 45-50 years ago, had priorities in the number of housing units. However, not in the quality of the infrastructure or the construction of the necessary piping systems. The lifespan of these piping systems is now borderline and due to a number of problems, they are being extensively replaced. This paper presents the results of the evaluation of the welded joints of old hot water piping systems and the condition after renewal.

2. Materials and Methods

- Base material of the original hot water pipe Pipe No. 1.

A cutout from a hot water pipe that had been in service for 45 years was used to evaluate the weld joint. The pipe used a P195GH base material according to EN 10217-5. This type of material is used for welded steel pipes for high pressure purposes. A cut-out was made from the original pipe with Ø 630 x 14 mm.

The chemical composition of the steel P195GH and its mechanical properties are presented in Table 1 and 2.

Table 1: Chemical composition of steel P195GH (wt. %).

C	Mn	Si	Cr	Mo	Ni	P	S	Fe
0,127	0,431	0,234	0,073	0,013	0,034	0,006	0,001	Bal.

Table 2: Mechanical properties of steel P195GH

R _m min [MPa]	R _e min [MPa]	A min [%]	KV min (-20° C) [J]
320	195	25	27

Fig. 1 shows a cutout of the original pipe, which was delivered after the hot water pipe was repaired. The welds on the original pipe are the subject of the evaluation. Their quality is assessed by means of non-destructive and destructive tests.



Fig. 1 Cutting of pipe No. 1 from the original hot water pipe

- Base material of the new hot water pipe Pipe No. 2

The new hot water pipe was made of P235GH material according to EN 10217-5. This steel grade is used to produce welded steel pipes for hot water and gas pipelines. The advantage of the material is its high-pressure capacity. The cut-out was made by oxygen-acetylene cutting from a new pipe after a repair of Ø 610 x 12,5 mm.

The individual chemical grades of P235GH steel obtained by spectral analysis can be seen in Table 3. The mechanical properties of the steel are given by the material data sheet in Table 4.

Table 3: Chemical composition of steel P235GH (wt. %).

C	Mn	Si	Cr	Mo	Ni	P	S	Fe
0,127	0,431	0,234	0,073	0,013	0,034	0,006	0,001	Bal.

Table 4: Mechanical properties of steel P235GH

R _m min [MPa]	R _e min [MPa]	A min [%]	KV min (-20° C) [J]
360	235	25	27

In Fig. 2, a cutout of the new pipe used in the repair of the new hot water pipe can be seen. The subject of the evaluation, as with the old pipe, was the welds. Their quality was evaluated by means of non-destructive and destructive tests.



Fig. 2 Pipe sample No. 2 from the new hot water pipe

The welding parameters used in the manufacture of hot water piping systems are in Table 5.

Table 5: Used welding parameters

Welding parameters	Pipe No.1	Pipe No.2	
Welding method	MMAW	GTAW	MMAW
Layer No.	1 - 2	1	2 - 3
Filler material	ESAB E-B 121	ESAB OK 12.61	ESAB OK 48.00
Diam. of the filler material [mm]	Ø3,2	Ø2,5	Ø3,2
Welding current [A]	110-140	90-120	110-140
Welding voltage [V]	16	16	24
Polarity	DC / +	DC / -	DC / +
Welding position	HL-045	HL-045	HL-045
Welding speed [mm/s]	1,02-1,36	0,63-1,03	1,02-1,36
Welding Machine	WTU 315	MINARC TIG EVO 200	
Weld	BW	BW	BW
Shielding gas flow rate Ar 4.6 [l]	-	16	-
Electrode [mm]	-	Wth - D 2,4	-

The procedure for filling the welds of pipe No. 1 and pipe No. 2 is shown in Fig.3 and 4.

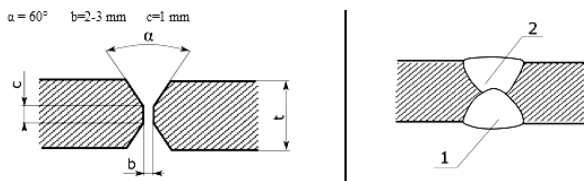


Fig. 3 Weld filling procedure of pipe No. 1

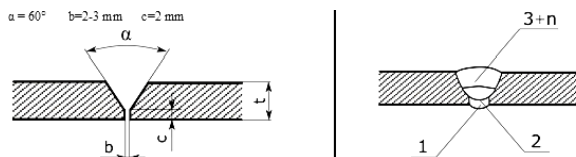


Fig. 4 Weld filling procedure of pipe No. 2

The following tests were carried out to assess the quality of the welded joints on pipe No.1 and pipe No.2.

- **Visual inspection of welds** - in accordance with EN ISO 17637. The evaluation of the weld quality standard is carried out according to ISO 5817 or ISO 6520. 500 lx illumination was used for the evaluation. A caliper, SPB-1A scale, SPM-1 scale, magnifying glass were used for weld evaluation. The evaluation also considered the quality of the adjacent areas of the welded joint within 15 mm of the weld edge. The evaluation also considered the size, elevation of the weld itself, presence of flash, weld drawing, surface pores, spatter, overflow, and weld root dimensions.
- **Capillary inspection of welds** - in accordance with EN ISO 23277. A colour indication method was used, using an aerosol indication kit from the manufacturer Diff - Therm. A BRE-S cleaner, BDR penetrant and BEA developer were used. 550lx lighting will be used. The presence of cracks, surface pores and inflammation on the surface and root of the weld will be assessed.
- **Tensile test of the welded joint in the transverse direction** - according to EN ISO 4136. The ZD 40 machine will be used to evaluate the load capacity of the welded joints and also the point of destruction of the test specimens. Test specimens marked 1 to 5 from the original hot water pipe - pipe No 1 and specimens marked 6 to 10 from the new hot water pipe - pipe No 2 are shown in Figure 5.
- **The bend toughness test** was carried out on ZD 40 on 10 test specimens in accordance with EN 5173. The test was carried out until a bending angle of 120° was reached. The test specimens before destruction are shown in Figure 6.

- **Weld microhardness evaluation** - according to EN ISO 9015-2 by Vickers on Shimadzu DUH 2. Indenter load used 98 g and load application time 15 seconds. The microhardness values on the specimens were measured at several locations of the weld joint such as ZM, TOO and ZK in terms of the schematics presented in Fig. 7. The microhardness was evaluated on transverse metallographic sections.
- **Metallographic analysis of macrostructure and microstructure** in accordance with EN ISO 17 639. The structures were documented on cross-sections using an Olympus BX FM light microscope. The samples were etched with 2% HNO3 solution.



Fig. 5 Samples before tensile test



Fig. 6 Samples before bend test

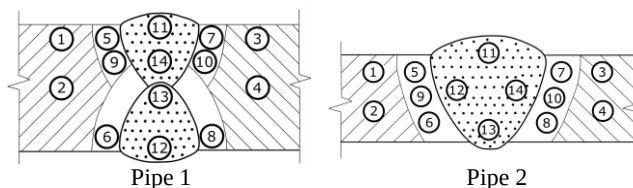


Fig. 7 Schematic of microhardness measurement

3. Results

From the results of the visual inspection, it can be concluded that the original welded pipe - pipe No. 1 had a spatter that extended along the entire length of the weld. The spatter is acceptable for the operation of the pipe. There is also a continuous inflammation visible in the weld area that extends down the side of the weld caterpillar. Surface pores are visible on the pipe. The pore size for a butt weld shall not exceed 3 mm. A detail of the welded caterpillar of the original pipe can be seen in Fig. 8. On the other side of the pipe Fig. 9, a continuously boiled root can be seen. After a visual inspection, the welded joint can be determined to be satisfactory. Even after several decades, this weld could still be serviceable. The cutout from the new hot water pipe No. 2 was satisfactory in its properties. The surface of the welded joint from the new hot water pipe is shown in Fig. 10. The root on the pipe was also evaluated and can be seen in detail in Fig. 11.



Fig. 8 Visual inspection of the weld surface – pipe No. 1



Fig. 9 Detail of the boiled root cutout from pipe No. 1



Fig. 10 Visual inspection of the new hot water pipe No. 2



Fig. 11 Detail of the boiled root cutout from pipe No. 2

The results of the capillary inspection confirmed the findings of the visual inspection. No non-compliant defects were detected using colour indication. The course of the capillary inspection is documented in Figure 12.



Fig. 12 Capillary weld inspection process

The tensile test results are rerecorded in Table. 6.

Table 6: The tensile test results of welds

Sample	R _m [MPa]	A80 [%]	Fracture	Place of fracture
1	489,94	27,7	Ductile fracture	BM
2	488,88	25,6	Ductile fracture	BM
3	485,29	25,4	Ductile fracture	BM
4	485,95	24,5	Ductile fracture	BM
5	492,81	23,3	Ductile fracture	BM
6	536,16	25,3	Ductile fracture	BM
7	530,23	25,1	Ductile fracture	BM
8	524,33	24,4	Ductile fracture	BM
9	531,36	24,7	Ductile fracture	BM
10	527,13	24,6	Ductile fracture	BM

* BM – base material

The samples after destruction are documented in Figure 13.

Based on the results of the bend test, it can be concluded that the test specimens marked 1 to 10 from the original pipe No. 1 and the new pipe No. 2 reached the prescribed angle of 120° without any damage. No damage and cracks or defects in the welded joint area, which could have occurred during the test, were visible.



Fig. 13 Samples after tensile test

The specimens after the bend test are recorded in Fig. 14.



Fig. 14 Test specimens after bend test

The measured microhardness values on the cross-sections are documented in the graphs of Figs. 15 and 16.

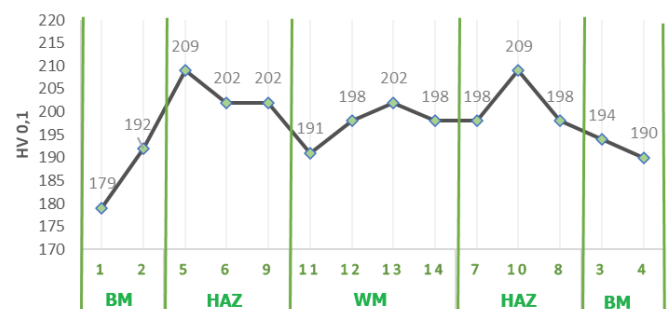


Fig. 15 Measured microhardness values on sample from pipe No. 1

From the above, on the butt weld specimen of the original hot water pipe, pipe No. 1 had a ZK microhardness region ranging from 191 HV to 202 HV 0,1. The highest microhardness was in the TOO region of 198 HV to 209 HV 0,1. The butt weld had the smallest values in the ZM region with a range of 179 HV to 192 HV 0,1.

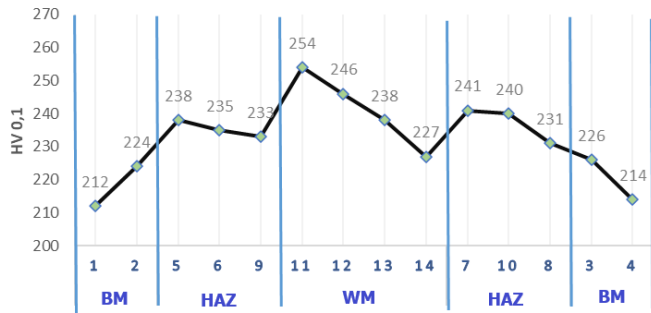


Fig. 16 Measured microhardness values on sample from pipe No. 2

From the weld specimen of the new hot water pipe, the ZK region achieved microhardness ranging from 227 HV to 254 HV 0,1. In the TOO region, the values ranged from 231 HV to 241 HV 0,1. The smallest values were achieved by the butt weld ZM region in the range of 212 HV to 226 HV 0,1. The results show that the ZK region achieved the highest microhardness values.

The results of the metallographic analysis are documented in Figs. 17 to 24. The macrostructure of the top and bottom of the weld from pipe No. 1 is recorded in Fig. 17. No internal defects were observed to be present in the weld. The structure has a characteristic spherical grain arrangement. An asymmetric heat affected region was observed. Figure 18 shows the structure of the base material of the P195GH steel tube. The structure is a row-like ferritic-pearlitic structure. In Fig. 19, a fine-grained ferritic-pearlitic polyhedral structure can be observed in the TOO and the transition to the base material.

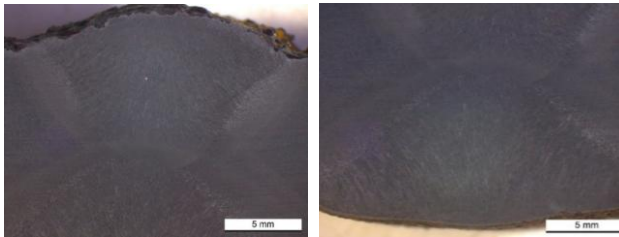


Fig. 17 Macrostructure of the top and bottom of the weld of the sample from tube No. 1

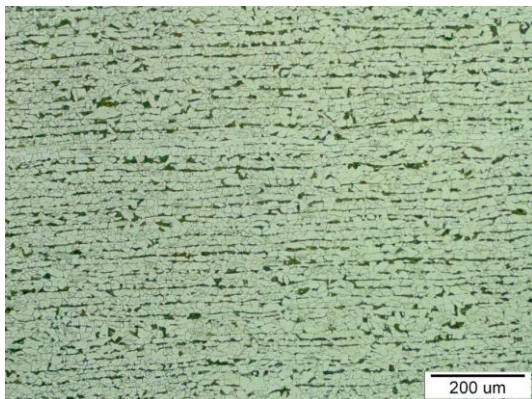


Fig. 18 Microstructure of P195GH steel base material

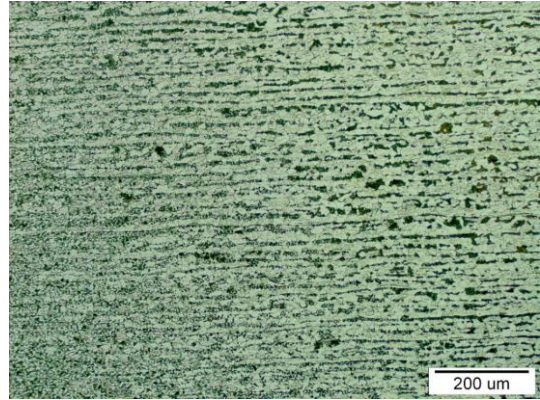


Fig. 19 Microstructure of the HAZ (heat affected zone) of pipe No. 1

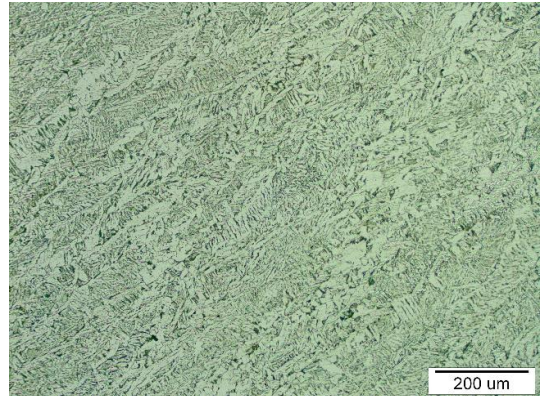


Fig. 20 Microstructure of weld metal from pipe No. 1

The structure of the weld metal from pipe No. 1 is shown in Figure 20. The structure is made up of the original austenitic grains. Acicular ferrite is excluded along the grain boundaries. Also visible are the columnar crystals from which the weld metal is formed. Figure 21 shows the macrostructure of the weld from pipe 2. No internal defects were observed in the weld. The structure has a characteristic coulumnar arrangement of grains. The structure of the P235GH pipe base material is shown in Figure 22. It is a linear ferritic-pearlitic structure. The presence of globular inclusions was observed in the structure. The microstructure of the HAZ is shown in Fig. 23. The structure is formed by acicular ferrite. Trapped polyhedral ferrite can also be seen in the region and inclusions were also observed. The weld metal structure in Fig. 24 was formed by Acicular and polyhedral ferrite.



Fig. 21 Macrostructure of the weld of the sample from tube No. 2

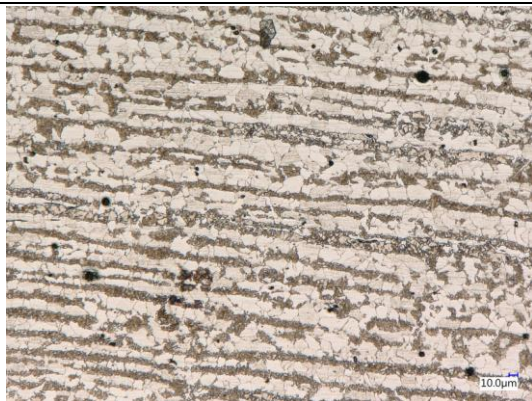


Fig. 22 Linear structure of P235GH base material



Fig. 23 Microstructure of the HAZ (heat affected zone) of tube No. 2



Fig. 24 Microstructure of weld metal from pipe No. 2

4. Conclusions

The paper presents part of the results from larger research focused on the quality assessment of welded joints made on hot water pipes. Due to the limited-service life of piping systems, the quality of construction execution 40 and 50 years ago, many piping systems are in a state of disrepair and their renewal requires considerable financial resources. Their construction is now being carried out under significantly stricter legislation than the original regulations. As part of the construction process, the quality of the welds is required to be checked by means of both non-destructive and destructive tests. The results of this inspection are part of the approval processes for the construction of these pipeline systems. They are indispensable for the development of WPQR production procedures.

Acknowledge

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Effect of production technique on a high silicon al-si-ni sintered alloy structure and properties

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Abstract: An experimental study was carried out to develop a technique for manufacturing a high silicon Al-Si-Ni sintered alloy with a coefficient of thermal expansion close to steels. A stage of hot forging was added to overcome porosity and improve silicon inclusions morphology to obtain better mechanical properties. Liquid and solid phase regimes of sintering were studied for the material made of the elemental components powders mixture as well as of a pre-alloyed powder. The impracticality of liquid-phase sintering modes is shown for this type of material primarily because of the tendency to liquid-phase exudation. It was proposed to carry out a solid-state vacuum sintering with subsequent hot deformation. Only the pre-alloyed powder is suitable for the technique; the other way, it leads to the coarse and sometimes, not equilibrium intermetallic inclusions will form in place of nickel particles because of the insolubility of Ni in solid Al. It is shown the possibility of successful plastic deformation of this material after sintering despite its extremely low ductility, which helps successfully overcome porosity and improve the characteristics of the microstructure of the material.

KEYWORDS: ALUMINIUM, SILICON, NICKEL, SINTERING, HOT FORGING, COEFFICIENT OF THERMAL EXPANSION

1. Introduction

Lightweight materials with the given physical properties are crucial for aerospace equipment production [1, 2]. For precise navigation devices, the coefficient of thermal expansion (CTE) is extremely important to be conformed to one of the contact materials such as steel, copper alloys etc. It is known that device details instability contributes up to 20 – 50 % of its total error [3].

Light materials on aluminium base are well proven owing to a number of their advantages such as corrosion resistance, plasticity, manufacturability, non-toxicity and relatively low cost [4]. However, CTE aluminium is rather high – $23.4 \cdot 10^{-6} \text{ K}^{-1}$ (for 20 – 100 °C) [5], which is significantly more than for steel ($11 - 15 \cdot 10^{-6} \text{ K}^{-1}$ [6]) or copper ($16.8 \cdot 10^{-6} \text{ K}^{-1}$ [7]). Thus, it seems to be an important task to reduce the CTE of aluminium-based materials with the mentioned destination. It is achieved mainly through alloying.

According to data from [4] Cr, Si, Be, and Cu reduce the CTE of aluminium. A low density of silicon and its efficiency in CTE decreases, making it primary for the purpose. However, we practically can't increase its content by more than 30 – 35 %, which would significantly reduce mechanical properties. But the CTE value obtained at such Si content is usually not enough. Nevertheless, it could be additionally reduced by an additional alloying with other elements. Among them, Ni is proven to be one of the most effective and harmless mechanical properties. 5 – 7 % of Ni is usually enough to obtain the desired CTE at 25 – 30 % of Si.

Casting technology is quite inadmissible for such high silicon materials with Ni mainly because of a wide range of solidification temperatures from ~800 – 900 °C (liquidus) to 557°C (solidus) [8, 9, 10]. Mentioned fact causes a significant liquation and a tendency for primary Si inclusions to grow large and coarse. So, the Si content in cast alloys often doesn't surpass 20 – 23 % [11]. Thus, powder metallurgy seems to be an appropriate manufacturing technique for the materials considered here.

However, there are questions of solid or liquid state sintering workability and efficiency as well as about the usage of a powder of a previously made alloy (then –pre-alloyed powder) or a mixture of the elemental powders. Some studies of liquid phase sintering for Al-Si, Al-Cu, Al-Si-Cu, and Al-Zn alloys [12 – 17] report its positive effects, such as porosity overcoming and better components consolidation. But all of them consider materials with Si content not more than 12 – 14 % and with other alloying elements, like Cu, Zn, or Mn. Thus, the production methods acceptable for the material considered here need a special study.

One of the ways to overcome porosity appearing in powder materials is hot deformation processing. However, Al-Si materials are known to be brittle and even hot deformation may need a special technique. Nevertheless, it is possible, and we had research experience in the area of Al-Si deformation-heat treatment study, and some results of structure and properties improvement were

achieved, but for cast alloys with lower Si content [18]. Here such a method is to be allied to the material produced through the powder metallurgy and with a significantly higher amount of Si.

2. Experimental material and technique

Approaches studied here are devoted to both a mixture of the pure initial components powders and a powder made of the pre-alloyed cast product. For the first one were used the following powders: Al 10 – 300 µm, Si 0.5 – 70 µm, and Ni 20 – 120 µm. The pre-alloyed powder was obtained through grinding in a planetary ball mill of the quickly cooled strips 30 – 50 µm strips. The obtained average particle size was $106 \pm 5 \text{ µm}$ with variation from 4 to 380 µm.

The average chemical composition of the powders according to the spectral analysis is given in Table 1.

Table 1

Chemical composition of the powders, wt. %

Material type	Al	Si	Ni	Fe	P
Pre-alloyed		31.32	6.51	0.068	0.097
Mixture of elemental powders	base	32.91	5.91	0.17	0.090

The powders were compacted under a pressure of 500 – 600 MPa. Steel covering shells were used due to it was assumed that they might optimize the subsequent deformation (forging) process, bringing it closer to the mode of all-around compression to reduce a brittle fracture probability. The outer diameter of the shells was 32 mm, the inner diameter was 28 mm, and the height, including the bottom and cover, was 37 mm. The bottom was welded before pressing. The top covers were set up after pressing and sintering.

The sintering occurs in a vacuum furnace. The temperature was set at 560 °C for the liquid phase sintering and 530 °C for the solid phase one. The sintering duration was 40 – 45 minutes for the liquid state one and 2 hours for the solid state one. Also, a preheating was used to 350 °C and kept at that temperature for about 30 minutes for the adsorbed water and hydrogen purification. The longer sintering time for the solid-state process was due to slower diffusion in the solids.

A special die was constructed to carry out the hot forging to provide a deformation regime close to all-around compression. The forging temperature was 520 – 540 °C.

Dilatometer measurements were performed on an induction quartz dilatometer assembled based on an induction transducer model 275-01 for measuring linear movements, the relative measurement error of which is $\pm 0.5 \text{ %}$.

Mechanical tests were carried out on cylindrical samples with a working part length $l_0 = 20 \text{ mm}$ and $\varnothing 3 \text{ mm}$ with deformation velocity $\dot{\epsilon}' = 2 \cdot 10^{-3} \text{ s}^{-1}$.

The study of the phase composition of the alloys was carried out on a DRON-4M diffractometer using $\text{Cu}_{K\alpha}$ and $\text{Co}_{K\alpha}$ radiation.

Electronic microscopy study and X-ray microanalysis of the present phases and structural entities composition were performed using TESCAN MIRA 3 device.

The microstructure of the samples was studied by optical microscopy. Metallographic studies were carried out on a NEOPHOT-2 optical microscope.

3. The results of the experiments and their discussion

Liquid phase sintering was found to lead to the liquid phase exudation both for materials from elemental components powders and from the pre-alloyed powder. Because of this reason porosity remained at the level of 20 – 25 % after such sintering.

The liquid phase exudation phenomenon, which is uncontrolled, is highly undesirable because of material loss and the chemical composition perversion. Also, it causes inner defects such as pores and hollows to appear, which couldn't be subsequently repaired. A liquid phase exudation is usually associated with poor wettability of the solid phase by the liquid. However, no information about its appearance during Al-Si alloys sintering was found in the literature. Usually, such a problem occurs for other systems, for example, Al-Sn [16].

The microstructure of the material after the liquid phase sintering is shown in Figure 1.

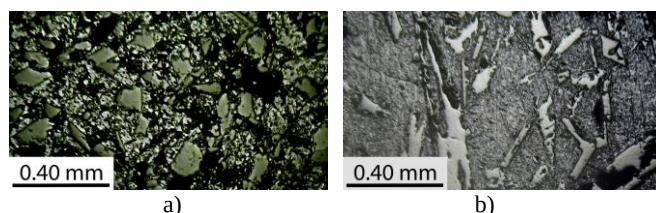


Figure 1 – The material structure after liquid phase sintering without subsequent deformation: a) from components mixture, $\times 40$; b) from the pre-alloyed powder, $\times 40$

A significant porosity seen from the structure remains after liquid phase sintering (Figure 1 – a) means that the liquid doesn't tend to fill the pores but leaks out. In the material made of the pre-alloyed powder (Figure 1 – b), we don't observe such a significant porosity on a micro scale, but rather macro defects (hollows). Silicon inclusions look similar to ones in the cast metal.

Because of the fact of liquid phase exudation, the decision was made to focus on the solid-state sintering with subsequent hot deformation. It should be noted that the task was to obtain a material of a certain composition which couldn't be changed significantly, so the composition correction to prevent the exudation was not an option.

A sample made of the powders mixture structure after solid phase sintering and subsequent hot forging at 530 – 540 °C is given in Figure 2.

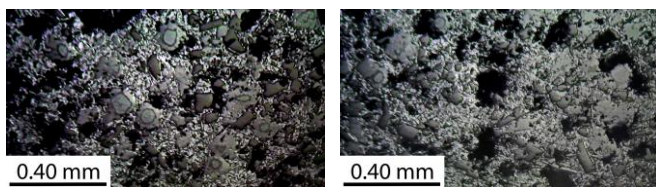


Figure 2 – The structure of the material made of the powders mixture after solid phase sintering and hot forging at 530 – 540 °C

Even after hot plastic deformation, such material remains porous. Silicon inclusions sizes are 50 – 70 μm . Also, specific entities having the shape of spherical inclusions with reams are observed in the structure. They are nickel particles surrounded by a layer of intermetallics, which was confirmed by X-ray micro spectral analysis. Figure 3 shows an SEM image of a part of such inclusion with the results of local analysis.

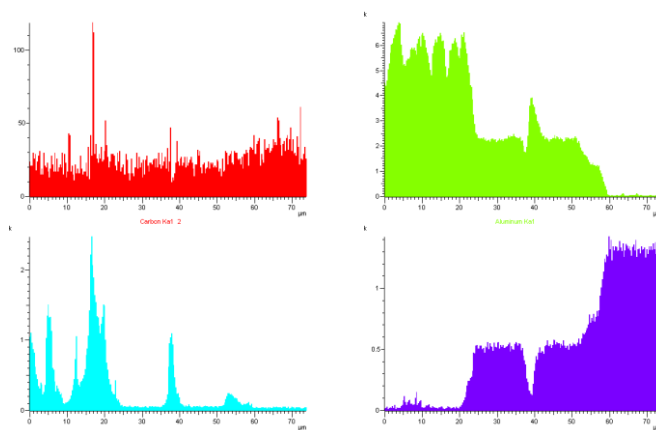
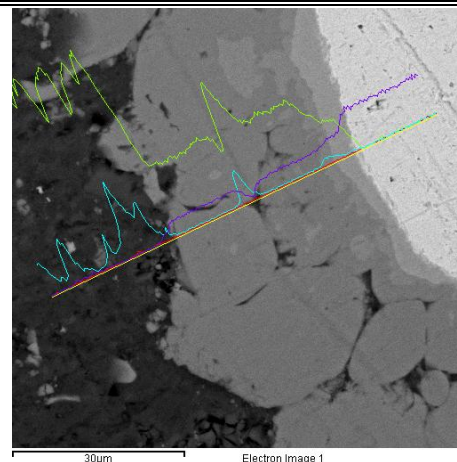


Figure 3 – Results of local X-ray microspectral analysis of the intermetallic structural component in a sample from a mixture of powders after solid phase sintering

The intermetallic shell is not dense, and it has cracks, but hot deformation, in this case, does not destroy such entities. Most of the intermetallic shell consists of NiAl_3 . On the side of nickel particles, there is a rather thin (2 – 3 μm) layer of other intermetallic with a greater Ni concentration, which is probably Ni_2Al_3 . Therefore, the nickel has not completely formed intermetallic compounds, so the final structure is non-equilibrium. The formation of such large-sized aggregates is because nickel almost does not dissolve in solid aluminium.

The best structure was obtained after the solid phase sintering and hot forging at 520 °C for the samples made of the pre-alloyed powder. Before the forging, the sintered samples were affected by preliminary hot deformation from sides with a setting degree of ~20 – 30 % at 510 – 520 °C. The structure of the material obtained in such a way is shown in Figure 4.

Silicon and intermetallic inclusions are dispersed (1 – 7 μm) and evenly distributed in the metal matrix. Thus, according to the obtained structure, such a manufacturing method looks the most acceptable for further production of the material.

Despite the shown disadvantages, the hot forging of the specimens after the liquid phase sintering was also studied here. In Figure 5 are shown the structures of the samples from the powders mixture and hot deformation. The porosity has decreased compared with the sintered state but remain. Those specific intermetallic entities observed after solid phase sintering were not found here.

The structures of the samples made of pre-alloyed powder after liquid-phase sintering and hot forging are shown in Figure 6. In the samples, affected by deformation at 530 – 540 °C without preliminary lateral hot deposition of the sample, areas with a typical cast structure are still observed – Figure 6 (a). Defects (pores) are also observed in these areas. However, most of the structure demonstrates equiaxed polyhedral silicon inclusions with a range of sizes from ~5 μm to ~60 μm and even more. Their average size, in this case, is $26.4 \pm 4.7 \mu\text{m}$.

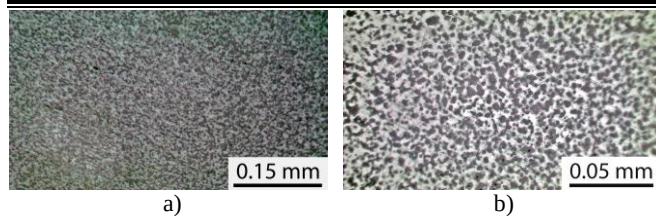


Figure 4 – The structure of the material from the pre-alloyed powder after solid phase sintering and hot forging at 520 °C: a) $\times 40$; b) $\times 500$.

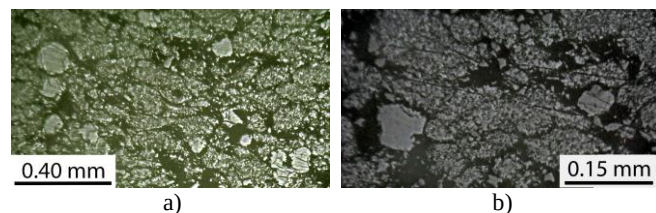


Figure 5 – The structure of the material from the powders mixture after liquid phase sintering and hot deformation at 540 – 550 °C: a) $\times 40$; b) $\times 100$.

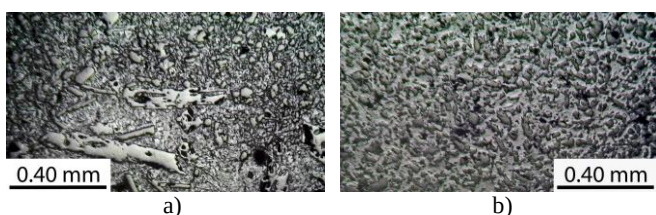


Figure 6 – The structure of the material from the pre-alloyed powder after liquid-phase sintering and hot deformation: a) forging at 530 – 540 °C, $\times 40$; b) forging at 520 °C with additional lateral pressing at 510 – 520 °C, $\times 40$

Lowering the forging temperature to 520 °C with adding the previous pressing from the sides at 510 – 520 °C leads to some structure improvement (Figure 6 – b). However, the silicon inclusions are still larger than the ones obtained after the solid phase sintering. Here were not found those cast-looking structures and significant pores. The inclusions sizes vary from $\sim 5 \mu\text{m}$ to $\sim 40 \mu\text{m}$ with an average size of $19.8 \pm 2.3 \mu\text{m}$.

X-ray diffraction quantitative phase analysis was carried out to clarify the phase composition of the material produced by different techniques. Its results are presented in Table 2.

The phases detected by this method were the solid solution of Al, Si and an intermetallic compound NiAl_3 . The lowest amount of NiAl_3 was found for types made from a mixture of powders. The possible and main reason for this may be the incompleteness of the intermetalide formation reaction. For one, after the liquid phase sintering, an additional reason may be the mentioned liquid phase exudation since NiAl_3 is part of the eutectic that melts and flows out at sintering temperatures. The most NiAl_3 was found in the samples made from pre-alloyed powder after the solid phase sintering. This amount looks very close to the equilibrium for the studied composition.

The method also allowed the estimation of the coherent scattering region sizes for the present phases of the studied material (table 2).

The sintering method has a valuable effect on the coherent scattering region sizes, especially for Al and Si-based phases. After solid-phase sintering, they are $\sim 1.5 \dots 2$ times larger than after sintering with the presence of the liquid phase. The effect is observed even though the specimens were made from completely different initial powders, which additionally indicates it.

Only for the samples made of pre-alloyed powder solid phase sintering was it possible to estimate yield strength. Such samples exhibited some but not significant ductility: total elongation was not more than $\sim 2 \%$. Such specimens exhibited the highest ultimate tensile strength and hardness.

Table 2

*Quantitative estimation of the phase composition of the studied specimen based on the X-ray diffraction results, weight %, and sizes of the X-rays coherent scattering regions, Å **

Specimen type	Al	Si	NiAl_3
From the powders mixture, after the solid phase sintering at 350 °C (~ 30 min) + 530 °C (~ 2 h) and forging at 530 – 540 °C	62.37 ± 0.83	26.09 ± 0.36	11.55 ± 0.17
	900	1020	660
From the pre-alloyed powder, after the solid phase sintering at 350 °C (~ 30 min) + 530 °C (~ 2 h) and forging at 520 °C	56.76 ± 0.51	24.83 ± 0.27	18.40 ± 0.31
	920	940	480
From the pre-alloyed powder, after the liquid phase sintering at 560 °C (40 – 45 min) and forging at 530 – 540 °C	59.92 ± 0.61	25.88 ± 0.30	14.19 ± 0.32
	520	560	460
From the pre-alloyed powder, after the liquid phase sintering at 560 °C (40 – 45 min) and forging at 520 °C with previous pressing from the sides at 510 – 520 °C	58.87 ± 0.44	26.78 ± 0.23	14.35 ± 0.20
	660	700	450
From the powders mixture, after the liquid phase sintering at 560 – 580 °C (~ 40 min) without forging	67.08 ± 0.74	22.59 ± 0.26	10.33 ± 0.15
	470	660	460

* - upper value – weight % of the phase; lower value – size of the X-rays coherent scattering regions, Å

The structure patterns obtained after different ways of manufacture reflect the mechanical properties. The data obtained through tensile tests and hardness measurements are presented in Table 3.

Table 3

Mechanical properties depending of the way of the material obtaining

No.	Samples' type	σ_Y , MPa	σ_U , MPa	Hardness, HB
1	From pure components mixture. Liquid phase sintering and hot forging at 530 – 540 °C.	—	81.3 ± 27.2	68.1 ± 5.3
2	From the pre-alloyed powder. Liquid phase sintering and hot forging at 530 – 540 °C.	—	116.6 ± 10.5	76.9 ± 4.5
3	From the pre-alloyed powder. Solid phase sintering and hot forging at 520 °C with preliminary hot deformation by pressing from sides.	220.7 ± 10.3	261.2 ± 20.9	95.8 ± 3.2
4	From the pre-alloyed powder. Liquid phase sintering and hot forging at 520 °C with preliminary hot deformation by pressing from sides.	—	139.7 ± 12.4	82.5 ± 5.7

All the other studied types of samples have significantly lower strength and hardness values, which are caused by larger and rough-shaped silicon inclusion in addition to porosity. Among them, the strongest were the samples made of the pre-alloyed powder after liquid phase sintering and forging at 520 °C with preliminary hot lateral deformation, which was almost nonporous. The highest ultimate tensile strength shown for such material was 145.4 MPa. Samples made of pure components mixture have the lowest strength and are very brittle. These facts additionally prove the benefit of using the pre-alloyed powder and the inexpediency of liquid phase sintering for the material.

The considered material also is to have a coefficient of thermal expansion (CTE) close to those of steel. To measure it, were carried out dynamometric tests. These are given in Table 4.

Table 4

CTE values of the material at the studied temperatures

Samples' type	CTE, K ⁻¹ · 10 ⁻⁶ at the temperature, °C					
	50	100	150	200	250	300
Cast state		13.2		14.2		
Liquid phase sintering and forging	10.51	12.95	13.18	13.49	13.82	14.13
Solid state sintering and forging	13.81	14.98	15.42	15.84	16.19	16.52
Solid state sintering and forging + annealing at 410 – 420 °C for ~1 hour	13.93	15.10	15.72	16.30	16.69	16.99

Because liquid phase exudation leads to an increase in silicon content, the samples after the liquid phase sintering have lower CTE values. For the specimens after the solid-state sintering and hot forging, its value at lower temperatures is $13.81 \cdot 10^{-6} \text{ K}^{-1}$, which is quite close to steel CTE. However, for low-alloyed steels, it is lesser – $11 \dots 12 \cdot 10^{-6} \text{ K}^{-1}$. Nevertheless, it is closer to it than ones of pure aluminium and cast Al-Si alloys. The closest CTE is observed for Cr and Ni alloyed stainless steels, high Cr instrumental steels, and special steels with Cr and Co. Annealing at 410 – 420 °C for ~1 hour after deformation might lead to some rise of CTE, especially for the higher temperatures.

4. Conclusions

An experimental study was carried out to find out an appropriate manufacturing method to produce the high silicone Al-Si-Ni alloy (25 – 30 % Si and 5 – 7 % Ni) with a close to steel CTE value. The hot forging stage was applied to obtain better mechanical properties. Liquid and solid phase sintering types were studied.

The best structure and mechanical properties values were obtained for the material made of the pre-alloyed powder using the following method: pressing (500 – 600 MPa) of the powder into steel shells, vacuum sintering (30 min at 350 °C then 2 h at 530 °C), lateral 20 – 30 % pressing at 510 – 520 °C, hot forging at 520 °C. Such a technique allows obtaining of a non-porous material with homogenously distributed fine spherical silicon inclusions with sizes of 1 – 7 µm.

It was found that liquid phase exudation during the sintering makes such a technique impractical for material production. Not only does it lead to uncontrollable distortion of the chemical composition and promotion of pores and hollows formation, but it also tends the silicon crystals in the liquid to grow, which further rough cast-like structures with significantly lower strength.

It was shown high insolubility of elemental powder mixture usage as the raw material for the solid-state sintering. It is because Ni doesn't solve in solid Al and tends to form rough intermetallic aggregates that could include non-equilibrium compounds. Instead, a pre-alloyed powder of the given composition would be a far better option. It could be obtained in different ways, for instance, by grinding a quick solidified strip.

It was found that the manufacturing method also has a significant influence on the sizes of the X-ray coherent scattering regions for the Al and Si phases. They appear ~1.5 – 2.0 times larger after the solid phase sintering than after liquid phase one, either for specimens manufactured from the powders mixture or the pre-alloyed powder.

Obtained as a result, this study material has high mechanical properties ($\sigma_Y \sim 221 \text{ MPa}$, $\sigma_U \sim 261 \text{ MPa}$, and hardness $\sim 96 \text{ HB}$), and its CTE is proven to be appropriate.

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Development of Software Application in the Evaluation of Erythrocyte Aggregation Studied with Microfluidic Device

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Abstract: The main focus of this research is on the elaboration of the combined experimental approach and image analysis (based on the specialized software environments) to the research of erythrocyte aggregation, which is evaluated by the microfluidic device BioFlux. To realize a precise evaluation of the erythrocyte aggregation index based on the obtained, during the experiment images, are applied image processing toolbox from ImageJ and an elaborated computer program in IntelliJ IDEA (integrated development environment) as well. The obtained results for the index of erythrocyte aggregation, based on the developed methodology, show a statistically significant difference between the two studied groups with preeclampsia and healthy pregnant women.

Keywords: MICROFLUIDIC DEVICE, SOFTWARE METHODOLOGY, IMAGE PROCESSING, ERYTHROCYTE AGGREGATION, PREECLAMPSIA

1. Introduction

In recent years, biomedical studies are a rapidly growing scientific field. One of the reasons for this is the usage of knowledge, procedures (algorithms), methodology, and technology, which are developed in Bioinformatics that is benefiting healthcare [1].

Bioinformatics covers obtaining important information and involves creating software programs based on various algorithms, data collecting, image processing and analyzing, and computer simulation [2].

Currently, different software instruments/programs are applied in the realization of image processing. In general, they can be separated into two major categories: the graphical user interface (GUI) and the command line interface (CLI) [3]. The first group of software instruments (GUI) allows biological images to be analyzed by using a graphical user interface with a set of ready-to-use options for image processing. These software instruments don't allow the creation of independent software applications by coding. This group of software environments (GUI), includes: ImageJ [4, 5], ICY [6, 7], CellProfiler [8, 9], etc. In the second type - CLI software, the researcher has to implement software commands (organized as algorithms and programs) to process and analyze images [3]. To this software tool group (CLI Tools) belongs environments such as Matlab (MATrix LABoratory) [10], LabVIEW Laboratory Virtual Instrument Engineering Workbench [11], IntelliJ IDEA [12], etc. No matter their interface, complex and structural differences, these categories are applied, in combination or separately, in scientific studies related to biological image processing and analysis [13]. The result is always oriented to support the process of studying and treating different diseases, such as preeclampsia – a pathological state, associated with abnormal placentalation, and excessive maternal inflammatory vascular response [14, 15].

Elevated red blood cell (RBC) aggregation is observed in women with metabolic disorders – such as preeclampsia [16]. For this pathological condition, increased erythrocyte aggregation is an established blood marker for systemic inflammation [14].

The RBCs can aggregate to realize two- and three-dimensional structures when suspended in solutions containing large plasma proteins (e.g. fibrinogen) or polymers (e.g., 70 kDa dextran). These structures are formed at stasis or when fluid shear forces are sufficiently low and can be dispersed by higher shear forces. The two-dimensional structures are formed by "face-to-face" contact of RBCs into linear arrays whose shape is similar to a stack of coins - rouleaux [17].

The extent of RBC aggregation at various RBC concentrations (hematocrit) and flow conditions are explored and quantified by various techniques. Indirect techniques, such as erythrocyte sedimentation rate (ESR), light reflection/transmission, ultrasonic, or electrorheology techniques are popular due to their practicality and low cost. Unequivocal data may only be provided by optical microscopy techniques, in which digital image analysis may be employed for an efficient interpretation of the information appearing in the images [18].

To study the effect of aggregation under static or flow conditions, different ways to calculate the erythrocyte aggregation index are proposed in the literature, providing information on the geometric characteristics of the aggregates (size and shape) and the extent of the aggregation phenomenon [18]. The main feature of erythrocyte aggregation, analyzed in studies with physiological hematocrits, includes the area covered by the cells compared to the total image area [19].

Microfluidic technologies enable the effective study of RBC aggregation [20]. By using a high-speed video system, associated with a microscope and micro-pump, it is possible to investigate *in vitro* erythrocyte aggregation in microchannels, under conditions similar to these in blood vessels [21, 22, 23].

In this study, the main focus of the authors is on the elaboration of the combined experimental approach and image processing and analysis for the evaluation of erythrocyte aggregation in the pathological condition - preeclampsia, which is studied by the microfluidic device.

2. Materials and Methods

The erythrocyte aggregation is studied using the air pressure-driven microfluidic system BioFlux. This system is a high-quality imaging platform for conducting in-flow rheological analyses [24]. The microfluidic system consists of BioFlux 200 electro-pneumatic flow control pump system; BioFlux microfluidic plates; inverted fluorescence microscope Lumascope 620; computer configuration with operational and analysis software. For evaluation experiments of the erythrocyte aggregation, BioFlux plates 24-well 0-20dyn/cm², containing 8 microfluidic channels with cross-sectional dimensions of 350 µm in width and 75 µm in height are used.

The human blood samples are collected from volunteer donors in accordance with ethical norms of medicine, and all of them gave written informed consent. Experiments are realized with peripheral blood samples from 4 healthy pregnant women – the control group, and 13 pregnant women with preeclampsia (PE). Blood samples are drawn by venepuncture and collected in 6 ml tubes with K₂EDTA

The erythrocytes are isolated by centrifugation of the whole blood (by centrifuge Universal 320 R, Hettich) at 1200 g for 15 min, and the yellowish supernatant (plasma and white blood cells) is discarded. A 40% hematocrit erythrocyte suspension is prepared (by centrifuge Haematokrit 200, Hettich). From this erythrocyte suspension, 10 µl is taken and added to 200 µl of Dextran 70 solution (at a concentration of 4 g/dl), to stimulate erythrocyte aggregation.

The microfluidic channels are filled with 200 µl of the prepared diluted erythrocyte suspension in Dextran 70. The erythrocyte suspension is perfused through the microfluidic channels at shear stress of 5 dyn/cm², for 5 min. After that, the flow in the channels is reduced to very low shear stress at 0,1 dyn/cm², for 5 min. Images of the formed erythrocyte aggregates along the entire visible length of each channel are taken using the fluorescence microscope, working in the Phase Contrast mode.

The obtained images of the erythrocyte aggregation, after usage of a microfluidic device are imported as input data in the software environment Image J [5], and after that the obtained results are further processed in an elaborated software program in IntelliJ IDEA [12].

For every image with erythrocyte aggregation, the number of aggregates is determined; the aggregate size is identified, and the total area of the erythrocyte aggregates, as well.

After the image analysis, the Erythrocyte Aggregation Index (EAI) of the two studied groups of pregnant women - healthy and with PE, is calculated by the formula [19]:

$$EAI = \frac{S_2}{S_1},$$

where S_2 (in pixels) is the total sum of areas of the aggregates, and S_1 (in pixels) is the total observed area of one visual field of the microscope.

Data analysis is accomplished by using the software packages Sigma Plot 11.0. All data are presented as means ± SD (standard deviation). The significance of differences between the two studied groups pregnant women-healthy individuals and with preeclampsia, are evaluated using a t-test or Mann-Whitney Rank Sum Test. Differences are accepted as significant at $p < 0,05$.

3. Results

From the images obtained with the BioFlux system, a difference between the aggregation of RBCs in healthy pregnant women and patients with PE, is observed (Fig. 1). The small single erythrocyte aggregates (rouleaux) are formed in the control group (Fig. 1A).

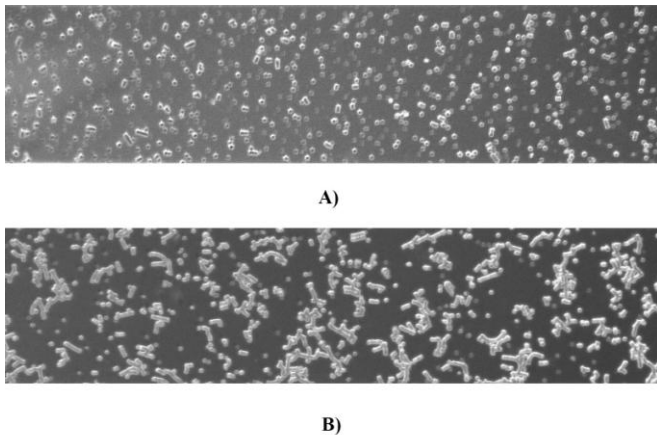


Fig. 1 Images of erythrocyte aggregates obtained with the BioFlux microfluidic system: A) healthy pregnant women; B) pregnant women with preeclampsia.

In the erythrocyte suspensions from women with PE large aggregates (rouleaux) are observed, that in addition form branched networks (Fig. 1B).

To determine the number and area of the obtained erythrocyte aggregates in both studied groups of pregnant women - healthy and with PE, a new method is developed to analyze the collected image data, using both experimental methodology and software environments - Image J [5] and IntelliJ IDEA [12]. The elaborated algorithm is shown in Fig. 2.

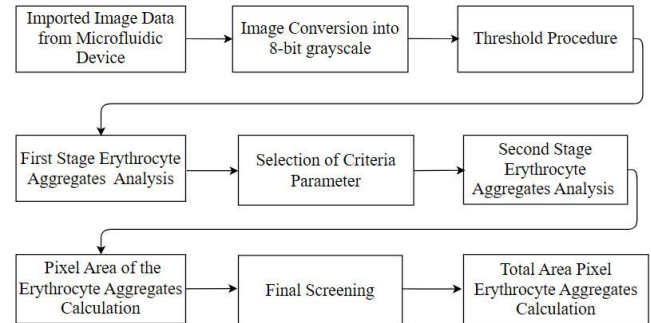


Fig. 2. Block-scheme of the algorithm for analyzing erythrocyte aggregation.

The image processing and analysis, illustrated in Fig. 2, contains the stages shown and explained below:

-Loading the image in the GUI environment of Image J [5].

-For the current study, to realize further image processing and analysis, the loaded image should be converted into an 8-bit gray style format. The applied software tool converts 16-bit and 32-bit images and stacks to 8-bits by linearly scaling from min-max values to 0-255. Depending on the current image selection, further calibration is required, to improve the brightness and contrast of the current image. For these types of 8-bit images, brightness, and contrast are changed by updating the image's look-up table (LUT) [13], so pixel values are unchanged.

- The next step of the image analysis consists of the erythrocyte aggregates localization (segmentation process) and contours determination of the erythrocyte aggregates (Fig. 3). Threshold procedure is applied to the selected image, dividing the background from the objects of interest (erythrocyte aggregates). These concrete default threshold steps are based on the algorithm IsoData. To indicate segmentation of the desired phase, this algorithm uses the white objects on a black background option.

The IsoData method is also known as *iterative intermeans*. It is an iterative procedure based on the iso-data algorithm [25]. This procedure divides the image into object and background by taking an initial threshold, then the averages of the pixels at or below the threshold and pixels above, are computed. The averages of those two values are computed, the threshold is incremented and the process is repeated until the threshold is larger than the composite average. This is explained with the following relation equation:

$$T = \frac{A_b + A_o}{2},$$

where T is the threshold ;with A_b - average background; A_o - average objects.

Based on the above-displayed threshold procedure, a segmentation of the image into areas of interest and background is realized. As a result of this processing, all objects (aggregates and non-aggregates) are recognized (Fig. 3).

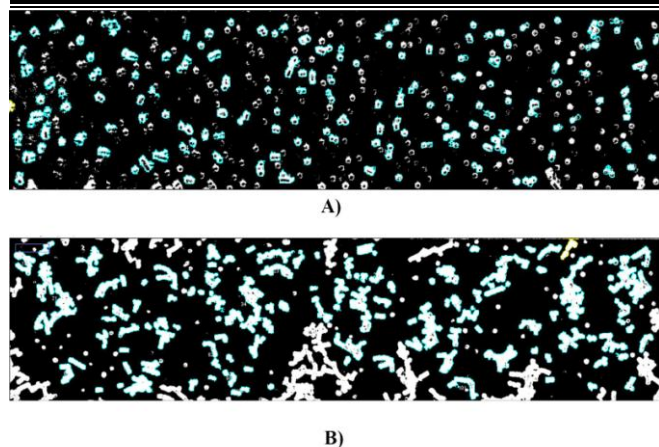


Fig. 3. Images of erythrocyte aggregates after application of threshold tool in Image J [12]: A) healthy pregnant women; B) pregnant women with preeclampsia.

-All objects from the image are marked. It is determined a criterion for the size in order to ignore the objects of non-interest. They are non-aggregates, that contain less than 3 erythrocytes and single erythrocytes.

-The next step is the evaluation of the area of every erythrocyte aggregate.

-If the obtained results satisfied the preliminary conditions in the final screening, the total data of the area of erythrocyte aggregates are collected and then it is processed in the elaborated by the authors of this study, a program in IntelliJ IDEA [12], where the total sum of their area is evaluated.

From the images obtained with a microfluidic system and by the developed methodology for analysis of the images, the erythrocyte aggregation index (EAI) is calculated. The total sum areas of all erythrocyte aggregates is divided by the total area of the entire visual field.

A difference between EAI for the control group and patients with PE is found (Table 1). The erythrocyte aggregation index EAI is increased in patients with PE (by 98 %; $p=0,009$), as compared to healthy pregnant.

Table 1. Mean values and standard deviations of EAI (Erythrocyte Aggregation Index) of the control group of healthy pregnant women and patients with preeclampsia.

Erythrocyte Aggregation	Healthy Pregnant n=4	Patients with PE n=13
EAI	0,064± 0,028	0,127 ±0,048 ^{###}

Mann-Whitney Rank Sum Test # $p<0,05$; ## $p<0,01$; ### $p<0,005$

4. Discussion

Image analysis is one of the most common methods for studying RBC aggregation [19, 23, 26 - 28]. It is possible to quantify the size and density of erythrocyte aggregates, by using this method [23, 27].

In the present work, the image processing methodology is developed and proved to be a powerful technique for characterizing RBC aggregation. This methodology for analyzing the images of erythrocyte aggregates is developed using the microfluidic system BioFlux. Software algorithm within the ImageJ software environment is created, and the obtained results are further processed developing a software application in IntelliJ IDEA. Under the current methodology, it is feasible to conduct a quantitative analysis of RBC aggregates under flow conditions. A key factor for applying the developed algorithm is good image quality. Since the methodology is based on image processing, it is very important to have a contrast between the background of the image and the aggregates, which have to be detected. The next

parameter to consider in the image processing technique is the threshold. Therefore, based on the obtained contrast, it is crucial to choose the optimal threshold at which all aggregates will be taken into account.

One of the most important step is the usage of the criterion for the separation of erythrocyte aggregates from non-aggregated and single cells. This criterion is related to determination of the size of the objects that are evaluated. In previous studies [23, 27, 28], no similar criterion is offered for analyzing images, obtained from a microfluidic device. This makes the developed methodology in this study very precise.

Finally, using the sum of the areas of the erythrocyte aggregates, the value of the erythrocyte aggregation index can be calculated. With this indicator, erythrocyte aggregation can be evaluated for various pathological conditions.

In the present study, erythrocyte aggregation is evaluated in women with preeclampsia and healthy pregnant women using the developed software and experimental methodology. The results show a statistically significant increase in erythrocyte aggregation in patients with PE in comparison to the control group (defined by healthy pregnant women). Increased abnormal erythrocyte aggregation is a biomarker of inflammation [14, 15].

The quantitative measurement of erythrocyte aggregation can be one valuable test to monitor the generalized inflammatory process detectable in the blood [14]. Therefore, the usage of the quantitative image analysis of erythrocyte aggregation is one prospective assessment of inflammatory states.

5. Conclusion

A new methodology, based on the developed software application and the experimental set-up has been proposed for evaluation of erythrocyte aggregation, studied with a microfluidic system. This methodology includes experiments with blood samples using BioFlux, image processing, and analysis of erythrocytes aggregation in two volunteer groups of pregnant women – healthy ones and with preeclampsia. The evaluation of the obtained images is realized under ImageJ and IntelliJ IDEA software environments., Quality and quantity criteria for control of the RBC aggregates are introduced, applying the image processing techniques. Applying the proposed methodology, statistically significant results for the index of erythrocyte aggregation are obtained, for patients with preeclampsia in comparison with healthy pregnant women.

Acknowledgment

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Polymer blends based on regenerated thermoplastics with low flammability

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Abstract: Composite materials based on regenerated thermoplastics of the polyolefin class with high parameters of stress-strain characteristics, which are highly resistant to open flame and combustion, have been developed. A synergistic effect was achieved by introducing a complex of modifiers into the matrix polymer, including regenerated particles of polytetrafluoroethylene, positioned as ultrafine particles (UPTFE), and residual products of the production of phosphate fertilizers – phosphogypsum (PG). The developed composite materials, processed by injection molding, make it possible to manufacture elements of metal-polymer rollers of belt conveyors used in the mining industry.

KEYWORDS: COMPOSITE MATERIAL, ENVIRONMENTAL IMPERATIVE, CONVEYOR BELTS, REGENERATED THERMOPLASTICS, SYNERGISTIC EFFECT

1. Introduction

The current trend of the mechanical engineering and other industries development is focused on achieving optimal parameters for the operational characteristics of functional products while ensuring increased safety requirements in combination with the implementation of the concept of the environmental imperative [1, 2].

In the nomenclature of composite materials used for the manufacture of functional elements of machines, mechanisms, technological equipment, a special position is occupied by composites with pronounced parameters of resistance to high temperatures and flames. This is due to the increasing consumption of such materials in mechanisms operated under specific conditions, when a violation of the regime can lead to consequences that are difficult to eliminate. These mechanisms include belt conveyors used in the mining industry to transport functional components of various composition, structure and technology for their subsequent processing [3].

The purpose of this work was to study the structural features and fire-retardant characteristics of polymer blends based on regenerated thermoplastics for their use in the manufacture of elements of belt conveyors used in the extraction of mineral components.

2. Materials and research methods

For the development of functional materials used in the construction of belt conveyors, thermoplastic polymers of the polyolefin class were used – original polypropylene (PP), regenerated production of Polish companies (PP(P)), regenerated production of JSC "Belvortopolimer" (PP(B)), regenerated production of JSC "Soligorsk Institute of Resources Saving Problems with Pilot Production" (SIRSPwithPP) (PP(S)) and a composite material based on polypropylene with low flammability PP(LF).

As modifiers of polymer matrices, waste products from the production of phosphate fertilizers were used – phosphogypsum (PG), ultrafine polytetrafluoroethylene (UPTFE), obtained by thermogasdynamic synthesis, produced under the trademark "Forum", regenerated elastomer (TPE) (Poland).

Mixed composites were obtained on an injection molding machine with a screw plasticizer under technological conditions characteristic of the processing of the base polymer.

The dispersed product Arguflame fr 30pp, which is part of the regenerated granulate based on polypropylene, was used as a flame retardant.

To assess the structural, deformation-strength and rheological characteristics of composites, the parameters of melt flow index (MFI), tensile strength parameters, fire and explosion hazard parameters and resistance to combustion, determined by standard methods, were used.

The evaluation of the effectiveness of the developed composite materials was carried out in the metal-polymer rollers used in the belt conveyors of SIRSPwithPP in the extraction and transportation of sylvinitic ores.

3. Results and discussion

Analysis of literature sources indicates a high efficiency of the use of polymer blends with low flammability modified with target components [4–6]. At the same time, the effectiveness of the modifier on the parameters of the composite structure and the characteristics of the stress-strain and thermophysical properties are largely determined by their dispersion. With a certain dimensional dispersed particles can exhibit the effect of the nanostate, which consists in the formation of adsorption bonds of the physical type, which lead to a qualitative change in the structure and parameters of the characteristics of the composite [4].

To modify polymer blends, we selected products of physicochemical synthesis for the production of phosphate fertilizers – phosphogypsum (PhG), which are technological waste products. These products with a particle dispersion of 1–50 μm, which are phosphorus-containing compounds, can have an effect similar to classical flame retardants [4, 5].

Another modifier was the products of thermogasdynamic synthesis of polytetrafluoroethylene, which are its highly dispersed particles, UPTFE. Our studies have shown that these products are not the result of synthesis, but are particles consisting of a polymer core surrounded by oligomeric compounds formed during thermal action on matrix particles of PTFE in an oxidation-free environment, due to the processes of degradation of the matrix with the formation of various oligomeric fractions, since the directed synthesis of PTFE macromolecules is not possible in the absence of a catalyst for the polymerization of low molecular weight fractions (monomeric). Therefore, the polymer-oligomeric structure of single particles of UPTFE is due to the processes of thermal degradation of matrix particles and is not associated with thermogasdynamic synthesis, which is possible only if the conditions of pressure and catalyst are met [6].

Studies of single particles of UPTFE have shown [5] that their central part has a size range corresponding to the nanostate, which significantly changes their activity in the processes of interaction with the polymer components of the composite material. At the same time, the presence of an oligomeric component in such particles significantly changes their structuring effect on the matrix. In addition, it is known that fluorine-containing compounds (polymer and oligomeric) are resistant to elevated temperatures and can act as flame retardants of composite materials based on thermoplastics [6].

As a polymer matrix in the development of composite materials with high resistance to temperature and low flammability, polypropylene and elastomer blends were used, obtained by thermomechanical combination of melts in the material volume of an injection molding machine with a screw plasticizer, which provides a sufficiently high homogeneity of the resulting product, which is necessary for its use as a composite material for the manufacture of structural elements of metal-polymer rollers of belt conveyors. The presence of modifier particles (UPTFE, PhG) in the polymer blends does not significantly affect the parameters of their rheological characteristics, allowing processing by injection molding (Table 1). At the same time, the studied composites have sufficiently high stress-strain characteristics (parameter of yield

strength (σ_y) is in the range of values of 17–30 MPa for composites based on regenerated polypropylene from various manufacturers PP(P), PP(B), PP(S) (Table 2).

The developed composite materials were evaluated in terms of resistance to high temperatures and flammability (Table 3) according to GOST 28157-2018 Plastics. Burning Resistance Methods, which is identical to ASTM D 3801 Standard Test Method for Measuring the Comparative Burning Characteristics of Solid Plastics in a Vertical Position. The essence of this method is to determine the burning and smoldering time of a vertically fixed sample.

Table 1. Rheological parameters of composite materials

No.	Sample composition	MFI parameter at temperature	
		MFI, g/10 min	T, K
1	PP(P)+0.5wt.% UPTFE	2.88	473
2	PP(P)+1.0wt.% UPTFE	2.80	473
3	PP(B)+0.5wt.% UPTFE	3.58	473
4	PP(B)+5.0wt.% UPTFE	3.80	473
5	PP(S)+0.5wt.% UPTFE	7.55	523
6	PP(S)+1.0wt.% UPTFE	17.10	523
7	PP(S)+5.0wt.% UPTFE	16.85	523
8	PP(LF)+0.5wt.% UPTFE	4.85	473
9	PP(LF)+1.0wt.% UPTFE	5.53	473
10	PP(LF)+5.0wt.% UPTFE	5.00	473
11	PP(B)+1.0wt.% PhG	4.80	483
12	PP(B)+5.0wt.% PhG	4.52	483
13	PP(B)+10.0wt.% PhG	5.15	483
14	PP(S)+1.0wt.% PhG	5.35	483
15	PP(S)+5.0wt.% PhG	2.20	483
16	PP(S)+10.0wt.% PhG	2.00	483
17	PP(P)+1.0wt.% PhG	3.95	483
18	PP(P)+5.0wt.% PhG	3.70	483
19	PP(P)+10.0wt.% PhG	4.33	483
20	PP(LF)+1.0wt.% PhG	6.70	483
21	PP(LF)+5.0wt. % PhG	7.25	483

22	PP(LF)+10.0wt.% PhG	6.00	483
23	PP(P)+10.0wt.% TPE + 5.0wt.% PhG	3.30	473
24	PP(P)+10.0wt.% TPE + 10.0wt.% PhG	2.78	473
25	PP(P)+20.0wt.% TPE + 5.0wt.% PhG	3.65	483
26	PP(P)+20.0wt. % TPE + 10.0wt.% PhG	3.62	483
27	PP(B)+10.0wt.% TPE + 5.0wt.% PhG	5.13	483
28	PP(B)+10.0wt.% TPE + 10.0wt.% PhG	5.15	483
29	PP(B)+20.0wt.% TPE + 5.0wt.% PhG	3.48	473
30	PP(B)+20.0wt.% TPE + 10.0wt.% PhG	4.53	473
31	PP(LF)+10.0wt.% TPE + 5.0wt.% PhG	6.15	483
32	PP(LF)+10.0wt.% TPE + 10.0wt.% PhG	2.95	483
33	PP(LF)+20.0wt.% TPE + 5.0wt.% PhG	5.08	483
34	PP(LF)+20.0wt.% TPE + 10.0wt.% PhG	7.20	523
35	PP(S)+10.0wt. % TPE + 5.0wt.% PhG	2.50	483
36	PP(S)+10.0wt.% TPE + 10.0wt.% PhG	2.33	483
37	PP(S)+20.0wt.% TPE + 5.0wt.% PhG	2.33	483
38	PP(S)+20.0wt.% TPE + 10.0wt.% PhG	1.40	483
39	PP(P)+10.0wt.% TPE	2.70	473
40	PP(P)+20.0wt.% TPE	2.03	473
41	PP(P)+30.0wt.% TPE	2.30	473
42	PP(B)+10.0wt.% TPE	3.25	473
43	PP(B)+20.0wt.% TPE	3.00	473
44	PP(B)+30.0wt.% TPE	2.95	473
45	PP (S) + 5.0wt.% TPE	14.95	523
46	PP (S) + 10.0wt.% TPE	15.65	523
47	PP (S) + 20.0wt.% TPE	2.98	523
48	PP (S) + 30.0wt.% TPE	9.75	523
49	PP(LF)+10.0wt.% TPE	4.43	473
50	PP(LF)+20.0wt.% TPE	4.15	473
51	PP(LF)+30.0wt.% TPE	4.05	473
52	PP(LF)+10.0wt.% TPE + 10wt.% PhG + 3.0wt.% UPTFE	3.78	483
53	PP(LF)+20.0wt.% TPE + 10wt.% PhG + 3.0wt.% UPTFE	5.90	483

Table 2. Stress-strain characteristics of composites

No.	Sample composition	Characteristic parameter for the sample					
		Physical yield strength. MPa	Deformation at physical yield strength. %	Strength at maximum force. MPa	Deformation at maximum force. %	Fracture strength. MPa	Deformation at fracture. %
1	PP(P)+0.5wt.% UPTFE	25.70	14.82	25.70	14.82	18.65	100.94
2	PP(P)+1.0wt.% UPTFE	23.45	11.45	23.45	11.45	14.53	100.41
3	PP(B)+0.5wt.% UPTFE	31.03	13.61	31.03	13.61	18.13	52.21
4	PP(B)+5.0wt.% UPTFE	27.88	7.21	30.25	13.60	30.25	13.60
5	PP(S)+0.5wt.% UPTFE	26.83	3.73	26.83	3.73	25.05	7.98
6	PP(S)+1.0wt.% UPTFE	25.40	3.64	25.40	3.64	23.38	9.56
7	PP(S)+5.0wt.% UPTFE	25.53	3.98	25.53	3.98	23.43	7.89
8	PP(LF)+0.5wt.% UPTFE	27.78	12.96	27.78	12.96	17.70	96.59
9	PP(LF)+1.0wt.% UPTFE	24.98	13.28	24.98	13.28	14.40	71.31
10	PP(LF)+5.0wt.% UPTFE	27.88	13.11	27.88	13.11	23.58	31.79
11	PP(B)+1.0wt.% PhG	30.20	9.07	31.98	14.61	31.98	14.61
12	PP(B)+5.0wt.% PhG	17.18	2.03	29.25	13.43	29.25	13.43
13	PP(B)+10.0wt.% PhG	27.30	10.11	27.63	13.63	27.63	13.63
14	PP(S)+1.0wt.% PhG	16.00	1.19	22.90	2.99	22.90	2.99
15	PP(S)+5.0wt.% PhG	18.08	1.53	21.65	2.67	21.65	2.67
16	PP(S)+10.0wt.% PhG	18.43	1.52	24.45	3.16	24.45	3.16
17	PP(P)+1.0wt.% PhG	23.33	21.90	23.33	21.90	17.30	104.04
18	PP(P)+5.0wt.% PhG	24.68	14.81	24.68	14.81	17.88	103.70
19	PP(P)+10.0wt.% PhG	18.78	16.00	18.78	16.00	13.20	58.21

20	PP(LF)+1.0wt.% PhG	21.24	12.53	21.24	12.53	19.56	23.80
21	PP(LF)+5.0wt.% PhG	22.68	14.11	22.68	14.11	20.55	25.02
22	PP(LF)+10.0wt.% PhG	23.38	10.94	23.38	10.94	21.73	22.58
23	PP(P)+10.0wt.% TPE + 5.0wt.% PhG	23.03	15.92	23.03	15.92	14.03	190.36
24	PP(P)+10.0wt.% TPE + 10.0wt.% PhG	19.23	17.26	19.23	17.26	16.30	226.55
25	PP(P)+20.0wt.% TPE + 5.0wt.% PhG	17.05	21.45	17.05	21.45	15.53	201.43
26	PP(P)+20.0wt.% TPE + 10.0wt.% PhG	16.83	21.30	16.83	21.30	14.50	160.49
27	PP(B)+10.0wt.% TPE + 5.0wt.% PhG	23.58	15.41	23.58	15.41	21.68	23.14
28	PP(B)+10.0wt.% TPE + 10.0wt.% PhG	25.10	14.75	25.10	14.75	23.08	24.34
29	PP(B)+20.0wt.% TPE + 5.0wt.% PhG	21.60	13.81	21.60	13.81	15.40	167.37
30	PP(B)+20.0wt.% TPE + 10.0wt.% PhG	10.20	2.70	14.50	11.23	14.50	11.23
31	PP(LF)+10.0wt.% TPE + 5.0wt.% PhG	25.03	16.92	25.03	16.92	21.80	36.00
32	PP(LF)+10.0wt.% TPE + 10.0wt.% PhG	17.10	1.87	25.85	9.95	25.85	9.95
33	PP(LF)+20.0wt.% TPE + 5.0wt.% PhG	20.95	15.93	20.95	15.93	17.10	203.09
34	PP(LF)+20.0wt.% TPE + 10.0wt.% PhG	19.43	18.56	19.43	18.56	15.63	64.81
35	PP(S)+10.0wt.% TPE + 5.0wt.% PhG	17.13	1.69	18.22	2.82	18.22	2.82
36	PP(S)+10.0wt.% TPE + 10.0wt.% PhG	20.20	2.21	20.20	2.21	17.13	7.90
37	PP(S)+20.0wt.% TPE + 5.0wt.% PhG	15.35	1.02	19.85	2.32	19.85	2.32
38	PP(S)+20.0wt.% TPE + 10.0wt.% PhG	15.33	1.02	19.85	2.32	19.85	2.32
39	PP(P)+10.0wt.% TPE	21.13	20.23	21.13	20.23	16.83	101.50
40	PP(P)+20.0wt.% TPE	16.48	22.92	16.48	22.92	13.93	103.96
41	PP(P)+30.0wt.% TPE	21.20	25.96	21.20	25.96	17.10	112.55
42	PP(B)+10.0wt.% TPE	29.23	13.96	29.23	13.96	18.13	62.28
43	PP(B)+20.0wt.% TPE	22.15	15.76	22.15	15.76	17.35	201.39
44	PP(B)+30.0wt.% TPE	22.20	21.30	22.20	21.30	16.75	201.51
45	PP (S) + 5.0wt.% TPE	15.70	1.18	22.68	2.99	22.68	2.99
46	PP (S) + 10.0wt.% TPE	17.33	1.60	22.63	3.81	22.63	3.81
47	PP (S) + 20.0wt.% TPE	18.23	1.68	22.98	3.31	22.98	3.31
48	PP (S) + 30.0wt.% TPE	18.35	14.16	18.35	14.16	14.40	287.33
49	PP(LF)+10.0wt.% TPE	23.75	12.88	23.75	12.88	11.30	124.04
50	PP(LF)+20.0wt.% TPE	26.23	15.57	26.23	15.57	10.00	67.83
51	PP(LF)+30.0wt.% TPE	28.43	15.25	30.33	18.45	14.32	121.45
52	PP(LF)+10.0wt.% TPE + 10wt.% PhG + 3.0wt.% UPTFE	23.20	12.66	23.20	12.66	19.70	36.32
53	PP(LF)+20.0wt.% TPE + 10wt.% PhG + 3.0wt.% UPTFE	19.78	15.09	19.78	15.09	15.88	118.66

Table 3. Parameters of resistance of composite materials to high temperatures

No.	The composition of the composite material	Characteristics parameter	
		Flame retardant	Flam-mability
1	PP(P)+0.5wt.% UPTFE	V-2	Average
2	PP(P)+1.0wt.% UPTFE	V-2	Average
3	PP(B)+0.5wt.% UPTFE	V-2	Average
4	PP(B)+5.0wt.% UPTFE	V-1	Average
5	PP(S)+0.5wt.% UPTFE	V-2	Average
6	PP(S)+1.0wt.% UPTFE	V-2	Average
7	PP(S)+5.0wt.% UPTFE	V-1	Average
8	PP(LF)+0.5wt.% UPTFE	V-0	Average
9	PP(LF)+1.0wt.% UPTFE	V-0	Average
10	PP(LF)+5.0wt.% UPTFE	V-0	Difficult
11	PP(B)+1.0wt.% PhG	V-2	Average
12	PP(B)+5.0wt.% PhG	V-2	Average
13	PP(B)+10.0wt.% PhG	V-1	Average
14	PP(S)+1.0wt.% PhG	V-2	Average
15	PP(S)+5.0wt.% PhG	V-2	Average
16	PP(S)+10.0wt.% PhG	V-2	Average
17	PP(P)+1.0wt.% PhG	V-2	Average
18	PP(P)+5.0wt.% PhG	V-2	Average
19	PP(P)+10.0wt.% PhG	V-1	Average
20	PP(LF)+1.0wt.% PhG	V-0	Average
21	PP(LF)+5.0wt.% PhG	V-0	Average
22	PP(LF)+10.0wt.% PhG	V-0	Difficult
23	PP(P)+10.0wt.% TPE + 5.0wt.% PhG	V-2	Average
24	PP(P)+10.0wt.% TPE + 10.0wt.% PhG	V-1	Average
25	PP(P)+20.0wt.% TPE + 5.0wt.% PhG	V-2	Average

26	PP(P)+20.0wt.% TPE + 10.0wt.% PhG	V-1	Average
27	PP(B)+10.0wt.% TPE + 5.0wt.% PhG	V-2	Average
28	PP(B)+10.0wt.% TPE + 10.0wt.% PhG	V-1	Average
29	PP(B)+20.0wt.% TPE + 5.0wt.% PhG	V-2	Average
30	PP(B)+20.0wt.% TPE + 10.0wt.% PhG	V-1	Average
31	PP(LF)+10.0wt.% TPE + 5.0wt.% PhG	V-2	Average
32	PP(LF)+10.0wt.% TPE + 10.0wt.% PhG	V-1	Average
33	PP(LF)+20.0wt.% TPE + 5.0wt.% PhG	V-1	Average
34	PP(LF)+20.0wt.% TPE + 10.0wt.% PhG	V-1	Average
35	PP(S)+10.0wt.% TPE + 5.0wt.% PhG	V-2	Average.
36	PP(S)+10.0wt.% TPE + 10.0wt.% PhG	V-1	Average
37	PP(S)+20.0wt.% TPE + 5.0wt.% PhG	V-2	Average
38	PP(S)+20.0wt.% TPE + 10.0wt.% PhG	V-2	Average
39	PP(P)+10.0wt.% TPE	V-2	Average
40	PP(P)+20.0wt.% TPE	V-2	Average
41	PP(P)+30.0wt.% TPE	V-1	Average
42	PP(B)+10.0wt.% TPE	V-2	Average
43	PP(B)+20.0wt.% TPE	V-2	Average
44	PP(B)+30.0wt.% TPE	V-1	Average
45	PP (S) + 5.0wt.% TPE	V-2	Average
46	PP (S) + 10.0wt.% TPE	V-2	Average
47	PP (S) + 20.0wt.% TPE	V-2	Average
48	PP (S) + 30.0wt.% TPE	V-1	Average.
49	PP(LF)+10.0wt.% TPE	V-1	Average.
50	PP(LF)+20.0wt.% TPE	V-1	Average.
51	PP(LF)+30.0wt.% TPE	V-1	Average.
52	PP(LF)+10.0wt.% TPE + 10wt.% PhG + 3.0wt.% UPTFE	V-0	Difficult
53	PP(LF)+20.0wt.% TPE + 10wt.% PhG + 3.0wt.% UPTFE	V-0	Difficult

The tests carried out made it possible to select for practical use several variants of composite materials based on regenerated thermoplastics with a combination of the necessary stress-strain characteristics, high manufacturability of processing into products by modern technologies and allowing the use of products from them in special conditions – with high requirements for flammability and exposure to an open flame. One of the areas of application of such materials is production of metal-polymer rollers of belt conveyors used in the mining industry. In addition to the requirements noted above, the materials for the manufacture of elements of metal-polymer rollers are also subject to requirements for high impact strength (Table 4).

Table 4. Charpy impact strength parameter for composite materials based on regenerated thermoplastics

No.	The composition of the composite material	Charpy impact strength parameter, kJ/m ²
1	PP(LF)+0.5wt.% UPTFE	1.39
2	PP(LF)+1.0wt.% UPTFE	1.44
3	PP(LF)+5.0wt.% UPTFE	1.49
4	PP(LF)+1.0wt.% PhG	1.12
5	PP(LF)+5.0wt.% PhG	1.74
6	PP(LF)+10.0wt.% PhG	1.34
7	PP(LF)+10.0wt.% TPE + 10wt.% PhG+3.0wt.% UPTFE	2.93
8	PP(LF)+20.0wt.% TPE + 10wt.% PhG+3.0wt.% UPTFE	5.92
9	PP(LF)	1.06

The presented data indicate that the developed materials based on polypropylene are significantly superior to the basic thermoplastic and can be recommended for the manufacture of elements of the metal-polymer roller of the belt conveyor used in special conditions.

4. Conclusion

The studies of the structural features and performance characteristics of composite materials obtained on the basis of regenerated thermoplastics made it possible to develop compositions with high parameters of stress-strain, technological characteristics and resistance to high temperatures and combustion. The use of the developed materials for the manufacture of elements of metal-polymer rollers of belt conveyors has shown the high efficiency of their practical application due to the reduction in cost and the return to the production of residual products of recycling of polymer components.

5. Acknowledgements

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Size distribution of the shape memory alloys powders obtained by pulsed electrical discharge in cryogenic liquids

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Abstract: The relatively simple in implementation the pulsed electrical discharge method as an alternative to the gas atomization was performed for the production of powders of shape memory alloys such as Ti-Ni-based, Ni-Al, Cu-Al-Ni, Ni-Mn-Ga as well as the glass-forming alloy Zr-Ni-Cu-Ti-Al and pure Ti. Several methods have used to analyze the powder size distribution spanning from ordinary sieving and laser diffraction to SEM studying. The most effective SEM method gave the information both for powder sizes distribution and morphology of submicron and micron sized particles. The analysis of SEM images has shown that there are several peculiarities both in the powder size distributions and cumulative distributions. That features reflect the action of several mechanisms of the powder particles formation sized between several micrometers to hundred micrometers. In particularly the powders particles size is in average more in powders obtained in liquid nitrogen than in liquid argon independent on the materials of powder. As well that the number of hollows particles sized between 10 and 50 μm is much more in powders obtained in liquid nitrogen.

KEYWORDS: PULSED ELECTRICAL DISCHARGE METHOD, POWDERS OF SHAPE MEMORY ALLOYS, POWDER SIZE DISTRIBUTION, LOGNORMAL DISTRIBUTION, CUMULATIVE DISTRIBUTION, HOLLOW PARTICLES

1. Introduction

The pulsed electrical discharge or spark erosion method of powder production is very attractive because of the exceptional simplicity of this method. Powders of several type of functional materials such magnetic alloys Co50Fe50, Ni-Mn-Ga, Fe83Ga17, Fe-Si-B [1, 2, 3], shape memory alloys Ti-Ni-Cu-Zr [4, 5, 6], Ti-Ni-Hf [5, 7], Cu-Al-Ni [8], Ni-Al [9], magnetic shape memory alloy Ni-Mn-Ga [3, 6] and bulk amorphous Zr-Cu-Al-Ni-Ti alloy [9], Ti-N [10] have been already prepared by this method. The vast of majority powder particles have sizes about several tens micrometers. However the size distribution is out of scope of the studying mostly although this characteristic is important for the understanding of sintering processes as well as indicates the peculiarities of powder formation mechanisms. The size distribution of powders can be investigated: a) by sieving; b) by granulometric analysis; c) by optical or electron microscopy. Sieving have been only providing preliminary information on the distribution of particles with sizes of tens of micrometers, which can be used for further analysis. So, this method was used to determine the part of the powder fraction of a brittle Ni-Mn-Ga alloy [6], obtained as a result of mechanical splitting of chunks in the spark erosion process in liquid Ar at significant electric discharge powers ($\Delta E/\text{imp}=6.2\text{J}$). Processing of Ni-Mn-Ga alloys in less energetic regimes caused a shift in the size distribution to the region of less than 70 μm . Thus the analysis by sieving becoming very coarse giving the low size resolution with lower limit of analyzing particles size in 40 μm . Therefore in our work [4], we analyzed the distributions of powder suspensions of Ti-N-Cu-Zr alloys by using the granulometric method (laser diffraction) on the CILAS 715 E482 apparatus. It was found that there are three regions in the size distribution of particles. The first area with a characteristic size of particles less than 1.5 μm can consist up to 25 wt% of powder. The second region, with particle sizes larger than 30 μm , is characterized by poor statistics. The third fraction, with a particle size below 30 μm , has a complex size distribution, which is caused by the sticking of powder particles, since the method analyzes a suspension. Therefore, the most effective method can be considered metallographic analysis using optical microscopy and SEM.

The aim of the current work was to carry out systematic investigation of the size distribution of powders particles obtained by electrical discharge using the analysis of the scanning electron microscopy images and find the correspondence between the distributions and powder formation mechanism.

2. Experimental

2.1. Powder preparation method

Elemental Ti and set of shape memory alloys Ni-Mn-Ga, Ti-Ni-Zr, Ni-Ti-Hf, Ni-Al as well as bulk amorphous Zr-Cu-Al-Ni-Ti alloy were

used as master alloy for the preparation of powders by pulsed electrical discharge method. This method also called as spark erosion method was described elsewhere in detail [4, 11, 5, 7]. The general principle of this method [12] involves the application of a heavy pulse current with duration of pulse 5-100 μs between two electrodes and a portion of pieces both prepared from the master alloy. Electrodes and chunks are immersed in a dielectric refrigerant inside a container. Liquid argon or nitrogen are used in our investigations. Many electrical discharges simultaneously appear between the chunks of material sized 3-5 mm during the pulse and form many conductive channels between the electrodes and chunks. When an electrical spark collapses molten particles and vapors of constituents are rejected from the boiling regions on the surface of small chunks and then quenched *in situ* in liquid refrigerant. Then the powders were kept in a vessel with cryogenic liquid during the day until all liquid evaporated after that the powder is collected.

Before powders elaboration alloys with the compositions $\text{Ni}_{49.0}\text{Mn}_{28.5}\text{Ga}_{22.5}$, $\text{Ni}_{46.7}\text{Ti}_{49.2}\text{Zr}_{3.4}\text{Cu}_{0.7}$, $\text{Ni}_{61.9}\text{Al}_{38.1}$ alloys were prepared by the induction melting, certain lots of Ni-Mn-Ga alloy and bulk amorphous $\text{Zr}_{57}\text{Cu}_{20}\text{Al}_{10}\text{Ni}_8\text{Ti}_5$ alloy – by arc melting. Ni-Al and Ni-Mn-Ga melts were molded in shape of sheets of $120 \times 25 \times 8 \text{ mm}^3$ while Zr-based melt was molded in shape of rods of 4mm diameter. The rods of $\text{Cu}_{67.9}\text{Al}_{27.5}\text{Ni}_{3.8}\text{Ti}_{0.5}\text{Cr}_{0.3}$, $\text{Ni}_{49.9}\text{Ti}_{40.3}\text{Zr}_{0.3}\text{Cu}_{0.1}\text{Hf}_{9.4}$ of 5 mm in diameter were melted and hot rolled by AMT NV (Belgium). Ti-Ni-Zr alloys were also subjected hot rolling at 900°C followed by homogenization at 1000 °C for 6 h. Ni-Al and Ni-Mn-Ga alloy were only homogenized because of their brittleness. Part of the Ti-Ni-Zr, Ni-Al, Ni-Mn-Ga sheets and Ti-Ni-Hf, Cu-Al-Ni, Zr-Cu-Al-Ni-Ti rods were ground into chunks of 2-4 mm. The remaining sheets, which were machined into two rectangular plates, or rods were used as electrodes. Liquid argon (LAr) was used as refrigerant for the Cu-Al-Ni, Ti-Ni-Hf, Zr-based, Cu-Al-Ni and Ni-Mn-Ga powders preparation. Liquid nitrogen (LN) was used for the Ti and certain lot of Ni-Mn-Ga powders preparation.

2.2. Powder particles sizes characterization

The morphology of particles and particle size distribution of Ni-Mn-Ga, Ti-Ni-Zr powder was preliminary studied by optical microscopy (OM) employing a Neophot-2 and Carl Zeiss Axvert 40 MAT conventional optical microscopes. The scanning microscopy (SEM) studying were carried out to reveal more details in the morphology and powder particles size distribution for all powders under consideration. A Hitachi S-2500 device, LEO 1530, JSM-6490LV, JAMP-9500F instruments were used for such kind investigation in addition to other structural studies described elsewhere in details [6, 5].

The advantage of this metallography approach is the ability to simultaneously analyze the morphology of particles, and in the case of SEM, the composition of individual particles. The analysis of the size distribution by OM method is ineffective due to the small depth of field of optical images (Fig. 1a). SEM analysis of cross-sections of powders particles mixed in epoxy resin (Fig. 1b) covered with a thin layer of Pd is much more effective.

The cross-section of powders mixed in epoxy resin can eventually pass through any place of the particle and for a spherical particle of radius R_0 theoretically any radius r of the cross-section in the range from 0 to R_0 can be observed on the images. Then, the average value of the radius and the square of the cross-section radius observed for a particle with a radius of R_0 is:

$$(1) \quad \langle R \rangle = \int_{-(R_0-\varepsilon)}^{R_0-\varepsilon} F(r) r dr \quad \langle R^2 \rangle = \int_{-(R_0-\varepsilon)}^{R_0-\varepsilon} F(r) r^2 dr$$

where $F(r)$ – the probability density of cutting a particle during the grinding process so that the observed cutting radius is r . It is also taken into account that it is impossible to observe cross-sections of particles with a radius smaller than some value ε of the order of the smallest particles of the abrasive material. Neglecting edge effects and assuming that all possible intersections are equally likely, and, therefore, the probability of crossing a particle at a distance x from the center is $F(x) = 1/2R_0$, we finally get:

$$(2) \quad \sigma^2 = \langle R^2 \rangle - \langle R \rangle^2 \approx 0.05R_0^2 \quad \sigma \approx 0.224R_0$$

As can be seen from the above, the measured values of the particle diameter will be underestimated by approximately 22%. Since the range of particle sizes was approximately the same for all powders, the illustration of the absolute error of diameter determination is shown only in Fig. 2a,c. The obtained estimate gives the maximum error of determining the radius, the absolute value of which is greater, the larger the radius. The relative statistical error of determining the number of particles is $1/\sqrt{N}$, where N is the number of particles of a given radius. Because of the small number of very large particles is analyzed in the photo, the relative statistical error for them was always

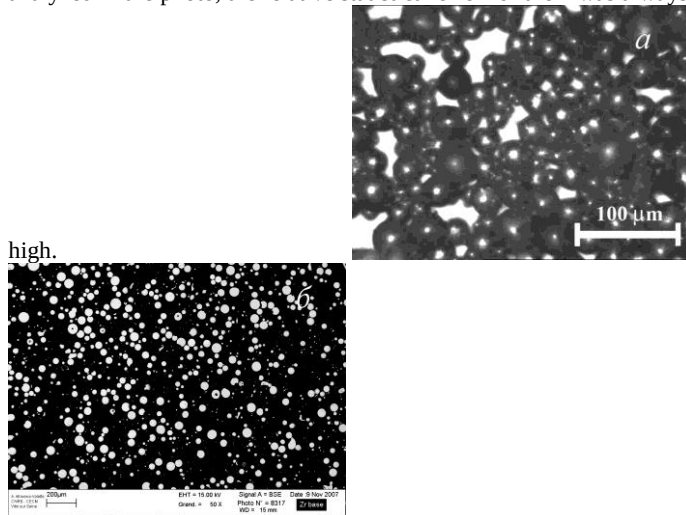


Fig. 1 a) Optical images of Ti-Ni-Cu-Zr alloy particles obtained in ethanol after washing in hexane and drying (according to [6]); b) cross-sectional images of Zr-Cu-Ni-Al-Ti alloy particles pressed in epoxy resin (backscattered electron SEM image).

3. Results and discussion

3.1. Distribution of micron particles by size

Typical histograms of the distributions of the number of powder particles by size $n(d)$ (Fig. 2a,c,e) illustrate that the vast majority of particles are smaller than 10÷20 microns; the number of particles

decreases sharply as their size increases. Around 30÷40 μm, there is a deviation from a monotonic decline. For Ni-Ti-Hf this is manifested as a maximum at ~35 μm. On Fig. 2b,d,f show the density distributions by volume fractions $f(d)$ of the powder particles. At the right edges of the density distributions by volume fractions, there is a significant scatter, and the statistical error can reach one order of magnitude with the value of $f(d)$ itself. This is due to the small number of large diameter particles in the samples.

3.2. Powder particles distribution interpretation

To estimate the average size of particles and their dispersion in the first approximation, it was assumed that the distribution densities satisfy the lognormal law. The distribution functions of the logarithm of the particle diameter then have the Gaussian distribution form:

$$(3) \quad f(\ln(D)) = \frac{1}{\sqrt{2\pi}\sigma^2} e^{-\frac{(\ln(D)-\mu)^2}{2\sigma^2}},$$

μ and σ^2 – are the mean value of the logarithm of the diameter and the variance of the lognormal distribution, which were determined as the empirical moment of the first and the central moment of the second order from the obtained data arrays of size N according to the formulas:

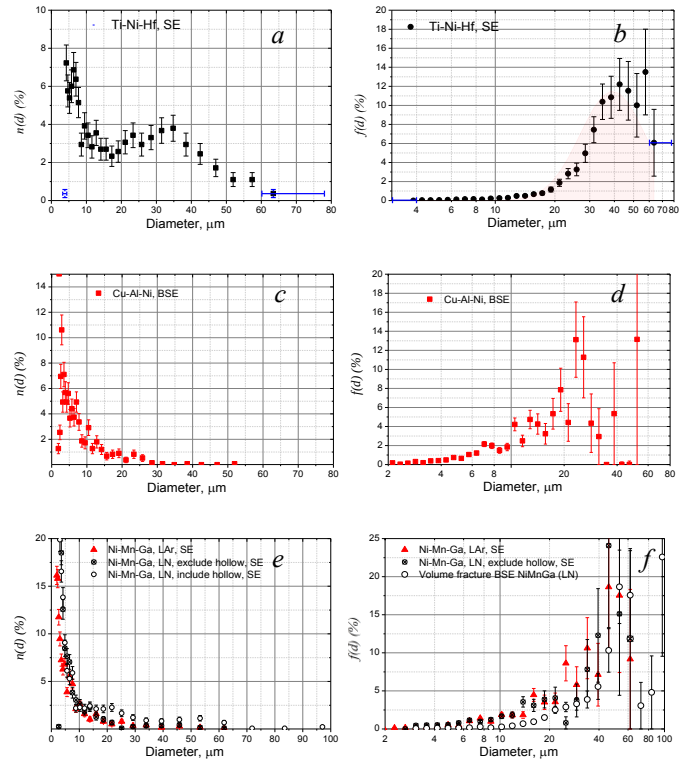


Fig. 2 On the left: the histograms of the distributions of the number of powder particles by size $n(d)$, on the right: the histograms of the density distribution of the volume fraction of particles by size $f(d)$ in powders of alloys Ti-Ni-Hf (a,b), Cu-Al-Ni (c,d), obtained by the pulsed electrical discharge method in liquid argon and nitrogen. In b), the shaded area represents the lognormal distribution that interprets the experimental distribution. For Ni-Mn-Ga alloys, distributions are given without taking into account (hollow excluded) and taking into account (hollow included) hollow particles. Marks BSE, SE mean distributions determined from the analysis of SEM images taken from backscattered and secondary electrons images, respectively. (a) and (b) show the errors in determining the radius of the particles for the two extreme points. For all other graphs, they are the same.

As can be seen from Fig. 2b, this distribution does not satisfy well the experimental one. To quantify the differences, the empirical values of the coefficients of asymmetry k and kurtosis γ were determined according to the formulas:

$$(5) \quad k = \frac{\mu_3}{\sigma^3} \quad \gamma = \frac{\mu_4}{\sigma^4} - 3,$$

where μ_i – are empirical central moments of the i_{th} order. μ , σ^2 , k , γ and the average value of the particle diameter defined as $\langle D \rangle = \exp(\mu)$ are given in Table 4.1

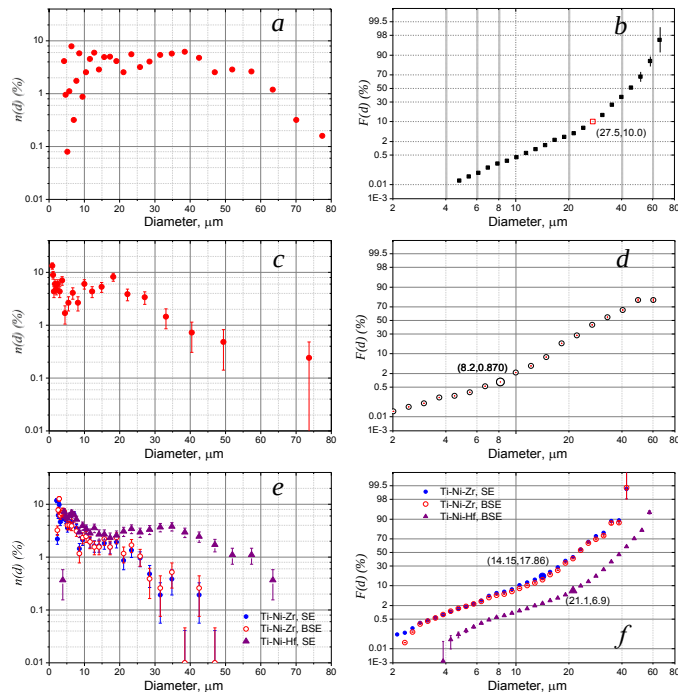


Fig. 3 The distributions of the number of powder particles by size $n(d)$ or the relative part of particles with diameter of d in a semi-logarithmic scale in powders of Zr-Cu-Ni-Ti-Al alloys (a), Ti obtained in a liquid nitrogen environment (c) and Ti-Ni-Zr alloys and Ti-Ni-Hf obtained in liquid argon (e). The corresponding figures on the right (b, d, f) show the corresponding cumulative distributions of the volume fraction of particles $F(d)$ with a diameter d smaller than the given one (here, the abscissa is on a logarithmic scale). The points on the distributions that separate two distinct sections of the distribution with the specified values of the characteristic diameter and the volume fraction of particles with diameters smaller than the characteristic are marked separately as (X,Y). BSE, SE marks the distributions determined from the analysis of SEM images taken in backscattered and secondary electrons, respectively.

3.3. Peculiarity of the powder particles and cumulative distribution and their relation with the powder morphology and the mechanisms of formation

The closest to the lognormal distribution are the distributions for Cu-Al-Ni and Ti – the coefficients of asymmetry and kurtosis for them were the smallest. The asymmetry of the distribution for all powders is to the left, and the peak is sharper. Only the distribution for Ti powder obtained in liquid nitrogen, volume fraction distribution by logarithm of diameter $\ln(D)$ is as close as possible to normal distribution. The trend is also obvious: the larger the average particle size, the more “tails” are stretched to the left (Ti-Ni-Hf, Zr-base, Ni-Al in LN, Ni-Mn-Ga in LN), and the average particle size of the powder obtained in liquid nitrogen environment, more than that obtained in liquid Ar.

It is convenient to present the distribution of $n(d)$ in a logarithmic scale along the ordinate axis (Fig. 3a,c,e, 4a,c,e). A local maximum is superimposed on the linear trend of the decline of the density of the

$$(4) \quad \mu = \langle \ln(D) \rangle = \frac{1}{N} \sum \ln(D_i) \\ \sigma^2 = \langle (\ln(D) - \mu)^2 \rangle = \frac{1}{N-1} \sum (\ln(D_i) - \mu)^2,$$

particle distribution, and in the case of the Ni-Mn-Ga powder obtained in liquid nitrogen, there are at least 2 of them. The rate of decline decreases starting from a certain value of the particle sizes. These features are due to different mechanisms of formation of particles of different sizes and morphologies.

The same features are also clearly visible on the graphs of the cumulative distribution of the volume fraction of particles $F(d)$

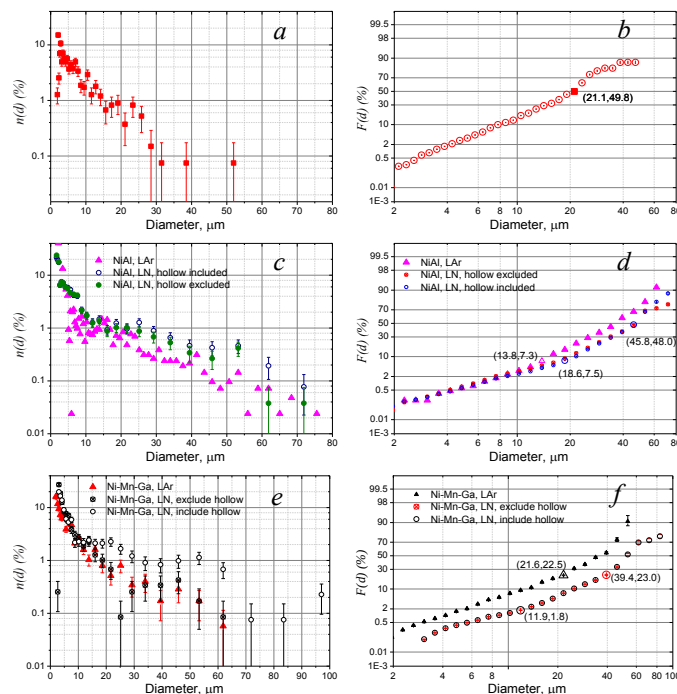


Fig. 4 The distributions of the number of powder particles by size $n(d)$ (or the relative part of particles with a diameter of d) on a semi-logarithmic scale in powders of Cu-Al-Ni alloys (a), Ni-Al, obtained in liquid argon and nitrogen (b) and Ni-Mn-Ga obtained in liquid argon and nitrogen (e). The corresponding figures on the right (b, d, f) show the corresponding cumulative distributions of the volume fraction of particles $F(d)$ with a diameter d smaller than the given one (here, the abscissa is on a logarithmic scale). The points on the distributions that separate the distinct sections of the distribution without taking into account (hollow excluded) and taking into account (hollow included) hollow particles are given.

(Fig. 4b,d,f). Here, the abscissa is presented on a logarithmic scale, and the ordinate is on a probability scale. The value of the latter represents the inverse cumulative Gaussian distribution, which, being presented along the ordinate axis on this scale, is a straight line. Thus, the plot of $F(d)$ presented in the mentioned coordinates is convenient for identifying the lognormal distribution. The graphs show a critical particle size D_{cr} , which separates two practically linear sections with different slopes (Fig. 3b,d,f, 4b,d,f). The value of D_{cr} , which varies from $\sim 8 \mu m$ for Ti-N and up to $\sim 28 \mu m$ for Zr-base, is presented in Table 4.1. The different slope of the linear sections should be interpreted as the presence of two types of particles with lognormal distributions, having different size dispersion. Smaller particles have greater dispersion. Note that their volumetric part, given their small size, is negligibly small.

In the function of the cumulative distribution of particles of Ni-Al and Ni-Mn-Ga powders obtained in liquid nitrogen (Fig. 4d,f), there is a

inflection, which is replaced by a linear section with the same slope as in the similar curves for powders obtained in liquid Ar. Starting from

Table 1 Empirical parameters of the distribution of particles of the powders particles. μ , $\sigma_{\ln(D)}$ is the mean value and variance of the hypothetical lognormal distribution. $\langle D \rangle$ is the average value of the particle diameter determined from the log-normal distribution; D_{cr} , D_h – critical values of particle diameters that distinguish different areas on the cumulative distributions of the volume fraction of particles $F(d)$ (see text)

Powder	$\mu = \langle \ln(D) \rangle$	$\sigma_{\ln(D)}$	$\langle D \rangle$ μm	D_{cr} μm	D_h μm	Asymmetry	Kurtosis	Comments
Ti-Ni-Hf	3.67	0.38	39.2	21.1		-1.52	3.90	left-side asymmetry, leptocurtic
Ti-Ni-Zr, SE	3.08	0.51	21.7	14.2		-1.10	1.62	left-side asymmetry, leptocurtic
Ti-Ni-Zr, BSE	3.12	0.49	22.6	14.2		-1.20	2.07	left-side asymmetry, leptocurtic
Zr-base	3.78	0.35	43.9	27.5		-1.23	3.68	left-side asymmetry, leptocurtic
Cu-Al-Ni	2.99	0.63	19.8	21.1		-0.56	0.31	left-side asymmetry, leptocurtic
Ti-N	3.53	0.56	34.2	8.2		-0.30	-0.13	left-side asymmetry, platykurtic
Ni-Al, LAr	3.55	0.55	34.7	13.8		-1.04	1.41	left-side asymmetry, leptocurtic
Ni-Al, LN	3.79	0.59	44.3	18.6	45.8	-1.36	2.64	left-side asymmetry, leptocurtic
Ni-Mn-Ga, LAr	3.47	0.61	32.0	21.6		-1.43	2.03	left-side asymmetry, leptocurtic
Ni-Mn-Ga, LN	3.98	0.52	53.7	11.9	39.4	-1.36	3.11	left-side asymmetry, leptocurtic

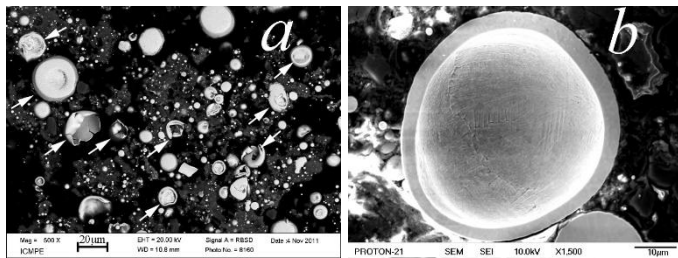


Fig. 5 SEM image of the micron fraction of powders obtained in liquid nitrogen, pressed in epoxy resin: a) Ni-Al powder. The arrows indicate hollow particles, b) cross-section of a hollow particle in Ni-Mn-Ga.

D_h size equal to $\sim 46 \mu\text{m}$ for Ni-Al and $\sim 40 \mu\text{m}$ for Ni-Mn-Ga, the slope of the linear section increases again. Therefore, in the interval from D_{cr} to D_h there is a concavity, the nature of which is related to the redistribution of the part of particles from this interval, hypothetically, due to the formation of hollow particles (Fig. 5). Their physical volume is less than the volume calculated based on their diameter, since the volume of the cavity in the medium is not taken into account. Some fraction of particles with diameter D "moves" into the zone of particles with diameter $D + \Delta D$. This explains why the average particle size of powders obtained in liquid nitrogen is larger than those obtained in liquid Ar. Let us mention that signs of such a dip are also observed in the distribution curves of powders obtained in liquid Ar. This indicates the universality of powder formation mechanisms, regardless of the production environment.

4. Conclusion

The density distribution $n(d)$ of micron particle sizes of the powders of shape memory alloys Ni-Mn-Ga, Ti-Ni-Zr, Ni-Ti Hf, Ni-Al as well as bulk amorphous Zr-Cu-Al-Ni-Ti alloy and Ti produced by electrical discharge has been studied by the analyzing SEM images. The distributions have a bimodal character, and the curves of the cumulative distribution of the volume fraction of particles $F(d)$ have inflections. This indicates the action of various mechanisms of the formation of particles sized between several micrometers to hundred micrometers that is reflected the particles morphologies too. In particularly the powders particles size is in average more in powders obtained in liquid nitrogen than in liquid argon independent on the materials of powder. As well that the number of hollows particles sized between 10 and $50 \mu\text{m}$ is much more in powders obtained in liquid nitrogen.

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Titanium-Based Porous Structures Produced by Powder Metallurgy Approach

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Abstract: Porous materials are very efficient in absorbing mechanical energy, for instance, in combined armor, in order to improve the anti-ballistic protection characteristics. In the present study, porous titanium-based structures were manufactured via powder metallurgy methods using titanium hydride (TiH_2) powder, which provided activated sintering, owing to dehydrogenation. The emission of hydrogen and shrinkage of powder particles on dehydrogenation also added a potential to control the sintering process and create desirable porosities. TiH_2 powder was sintered with additions of ammonium as pore holding removable agents. The microstructures and porosities of sintered dehydrogenated titanium with different concentration ammonium were comparatively studied. Mechanical characteristics were evaluated using compression testing with strain rates varying from quasi-static to high levels. All testing methods were aimed at characterizing the energy-absorbing ability of the obtained porous structures. The desired strength, plasticity and energy-absorbing characteristics of porous titanium based structures were assessed, and the possibilities of their application were also discussed.

Keywords: TITANIUM HYDRIDE, POROUS MATERIALS, MICROSTRUCTURE, POWDERS.

Introduction.

Metallic materials with controlled porosities are characterized by a number of unique physical and mechanical properties, due to which they are widely used in various fields—from filters to impact energy-absorbing systems [1–2]. A wide range of different technological approaches are used to obtain such materials: foaming of liquid metals, laser printing with metallic powder, powder metallurgy method including employment of removable space holders [2–4]. The last method, powder metallurgy with space holders, is the simplest and most versatile, allowing one to obtain materials with porosities of more than 80%, high specific strength, and sufficient strain values when tested with different deformation rates. Concerning titanium and titanium-based porous materials, early works were devoted to the production of porous titanium using NaCl as a space holder [5,6]. These studies were carried out either on hydrogenated/dehydrogenated titanium powder [5] or globular powder [6] sintered at relatively low temperatures (780 °C), which correlated well with the properties (strengths) of cancellous bones and was promising for use in implants. The works in which not only NaCl was used as a space holder together with aluminum powder ensuring the formation of highly porous intermetallic compounds of the Ti–Al system should also be noted [7]. The mechanical behavior of porous materials is determined with specified parameters of porosity, including total volume content, size of pores, and uniformity of their distribution. Thus, to achieve desirable strengths, ductility levels, and dumping capacities, the parameters of pore structures should be strictly controlled. The aim of the present work was the fabrication of highly porous titanium-based materials in employing earlier developed powder metallurgy approaches based on the employment of titanium hydride instead of pure titanium [8] to obtain desirable porous structures and investigations of the mechanical behaviors of produced materials under conditions of quasi-static and dynamic compression in order to evaluate their potential for energy absorption.

Materials and experimental procedure.

Powder metallurgy approach employing titanium hydride TiH_2 as raw powder [8–9] were tested in the present study to produce porous titanium-based materials. To achieve increased porosities, titanium hydride powder with a particles size of $<100 \text{ }\mu\text{m}$ (Figure 1a) was blended with ammonia carbonate (NH_4CO_3) (Figure 1d). Corresponding blends (Table 1) were compacted at room temperature at 150 MPa pressure and sintered using specified regimes to achieve dehydrogenation of titanium hydride,

consolidation of titanium particles, and removal of space holder agents. Porous commercially pure titanium (CP-Ti) was produced using ammonia carbonate (NH_4CO_3) as a removable space holder, which could be removed by annealing of green compacts at 150 °C in open air before vacuum sintering.

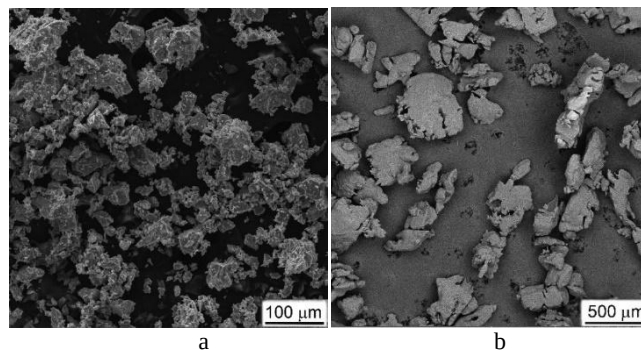


Fig.1 Microstructures of initial powders: (a) TiH_2 , (b) ammonia carbonate (space holders).

Cylindrical samples with gauge lengths and diameters of 10 mm for direct impact Hopkinson pressure bar (DIHPB) and quasi-static compression (QSC) tests were cut by the electric discharge machining technique. The first tests were performed with the installation shown in Figure 2; the second—using the Instron 8802 strength machine. Young's, Shear moduli, Poisson's ratios, and Damping capacities of materials were measured with the Resonance-Frequency-Damping Analysis (RFDA) system (IMCE, Genk, Belgium), using the Impulse Excitation Technique (IET), following the ASTM E1876-15 protocol. Material microstructures before and after tests as well as the specimens' fractures were studied using scanning electron microscope (SEM), Vega 3 (Tescan, Czech Republic), equipped with energy dispersive X-ray (EDX, Bruker, USA) spectroscopy allowing the measurement of the chemical composition of materials. Phase compositions of the specimens and their crystalline structure measurements, including texture analyses, were performed using an X-ray diffraction (XRD) Ultima IV (Rigaku, Tokyo, Japan) system. The gas content within the sintered specimens was measured using a gas analyzer ELTRA OH900 (Haan, Germany). Instron 8802 strength machine. Young's, Shear moduli, Poisson's ratios, and Damping capacities of materials were measured with the Resonance-Frequency-Damping Analysis (RFDA) system (IMCE, Genk, Belgium), using the Impulse Excitation Technique (IET), following the ASTM E1876-

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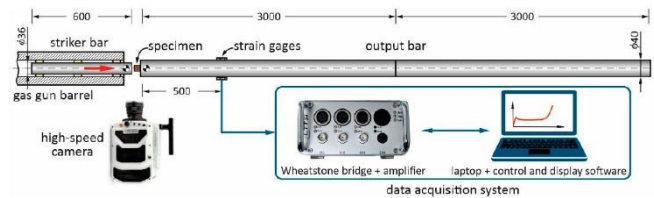


Fig. 2 The schematics of the forward DIHPB system used in the present work

Dynamic compression tests were performed using the DIHPB technique, represented by the forward configuration, which ensured the deformation of the specimens to large strains (up to densification in the case of cellular solids) [16,17]. The schematic drawing of the forward DIHPB set-up is depicted in Figure 2. The used solution of the DIHPB arrangement was similar to the one presented in [18,19]. The currently used laboratory stand consisted of a striker bar with a diameter of 36 mm and a mass of 4760 g, launched from a gas gun and a 6 m long output bar, i.e., two 3 m long pressure bars with a diameter of 40 mm and a data acquisition system. The specimen was put at the front of the output bar, and the 600 mm long striker bar could directly impact the specimen. The striker bar and output bars were made of the same material, i.e., C45 steel with a yield strength and elastic wave velocity equal to 735 MPa and 5140 m/s, respectively. To determine force/stress on the loading surface of a specimen, a pair of strain gages placed 500 mm from the front end of output bar was used to record transmitted signals. The position of the strain gages and the length of the output bar were chosen in order to avoid wave superposition.

A Phantom V1612 high-speed camera (Vision Research, Inc., Wayne, NJ, USA) was used to measure the shortening of the specimens, the compression rates, and to identify failure modes, as well as to confirm that the striker bar kinetic energy was sufficient to provide a near-constant compression velocity of specimen up to a nominal strain of at least 0.5. High-speed video images were recorded with a resolution of 512 × 208 pixels and a frame rate of 110,000 fps. To ensure high measurement accuracy based on video images, crush test markers and specialized TEMA Classic software version 4.6-002.64 (Image System AB, Linköping, Sweden) were used.

Results and discussion.

Typical initial microstructures of the studied porous materials are presented in Figure 3. It is clearly seen that all the sintered materials are characterized mostly by open porosities, which were most developed in the case of 75% ammonia carbonate (AC) as space-holding. In this case, the sizes of the formed pores (~100 μm) completely inherited the sizes of the initial material (Figure 3, and compare with Figure 1b). In addition to large pores in the locations of space holders, there is a multiplicity of small (less than 20–30 μm) pores between the consolidated dehydrogenated titanium particles. The application of ammonia carbonate (AC) as a space holder caused wide variations in pore sizes and total porosities (Figure 3 1), depending on the amount of pore holder in the initial blends.

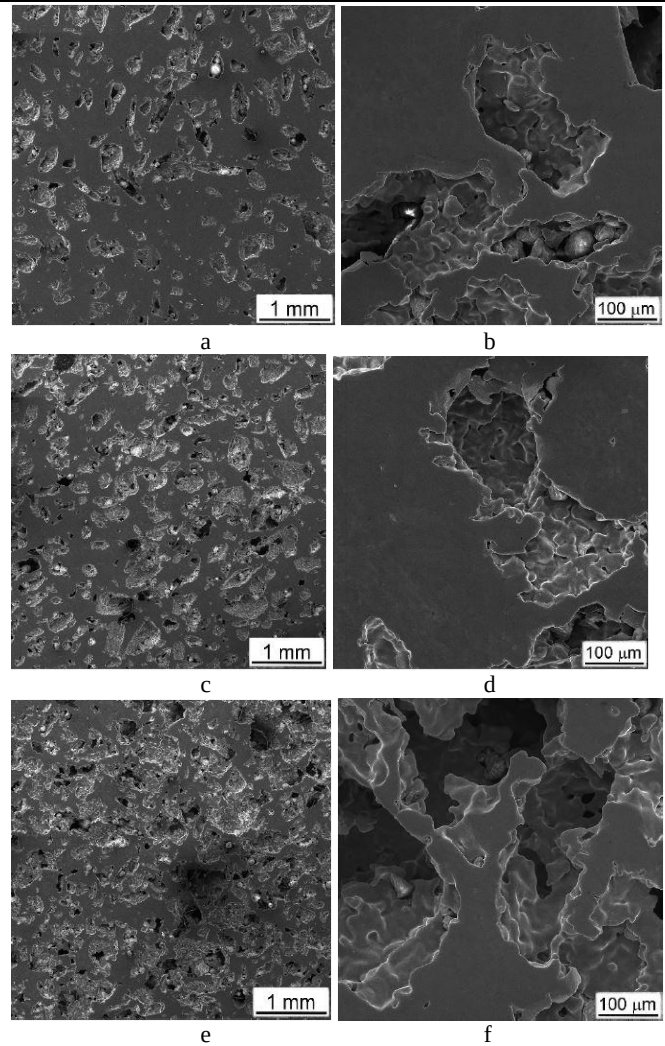


Fig.3. Microstructures of porous materials using ammonia carbonate (AC) space holders: (a,b) 25%, (c,d) 50% AC, (e,f) 75% AC.

The nominal (apparent) stress–strain curves of the porous samples were obtained with strain rates ranging from 10^{-3} to 10^{-1} s^{-1} . The representative stress–strain curves for produced porous materials under quasi-static loading are presented in Figure 4.

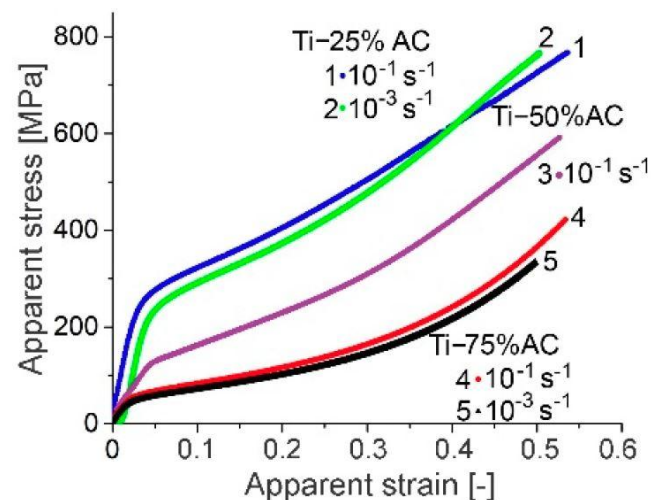


Fig.4 Quasi-static compression stress–strain curves of porous materials

Porous titanium obtained with ammonia carbonate (AC) showed more promising results (Figure 5c). In most cases, the stress–strain curves were smooth and typical for porous materials, i.e., a linear-like elastic stage appeared initially up to the yield stress level, followed by a period of long strain hardening, collapsed porous regions, and—in the case of Ti-50%AC and Ti-75%AC materials—an onset of densification. However, the stress–strain curve profile of the Ti-25%AC material was similar to the compression curve of the solid material (Ti-25%AC yield point was 232 MPa and it was lower by approximately 23% than the yield strength of commercially pure titanium), whereas other materials exhibited mechanical behaviors similar to open-cell foams (type I) or additively manufactured regular cellular materials [10]. At this point, it should be noted that the relative densities of Ti-50%AC and Ti-75%AC were 0.51 and 0.43, respectively; thus, they were higher than the upper value of the relative densities attributed to foams (according to the work [11]). Foam is defined as a cellular material, whose relative density ranges from 0.01 to 0.3). Hence, no plateau stage was observed, wherein stress was relatively constant over a large strain range. Instead, an increase in nominal stress was observed, which was associated with the effect of the strain hardening of the base material. Nevertheless, the visible positive slope of the nominal compression curve after exceeding the yield point was not only the effect of strain hardening but also the increase in the compression force, resulting from the increase in the specimen's cross-section, especially in a material with low porosity, such as Ti-25%AC.

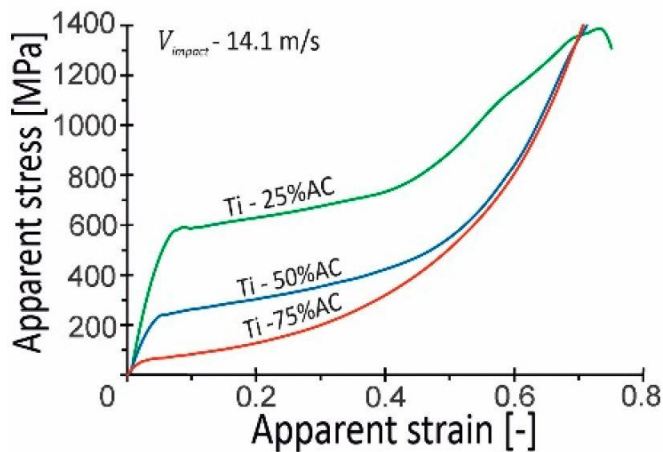


Fig.5 Representative dependencies between apparent stress and strain for porous specimens tested at initial impact velocity of 14.1 m/s:

To ensure that the specimen compression velocity under DIHPB conditions was relatively constant, the initial impact velocity was increased to 14.1 m/s on average. As a result, the relative shortening of the specimens for a 20% drop in the impact velocity was higher and amounted to 0.38 and 0.64 for Ti-25%AC and Ti-75%AC, respectively. Thus in the case of the Ti-75%AC material, the specimen crushing took place at a relatively constant velocity for more than half of the entire deformation process. The dynamic stress–strain dependencies for the porous specimens tested at a 14.1 m/s compression velocity are presented in Figure 5. The obtained compression curves are nominal and were calculated on the basis of the initial cross-section of the specimen. These representative stress–strain curves are derived from a dataset of at least three experiments, with a scatter of results less than 5% for all materials. For the assumed level of initial impact velocity, i.e., 14.1 m/s, the corresponding average deformation rate is about 1300 s^{-1} for a 20% decrease in compression velocity after impact.

After the initial stage of compression, during which the samples deformed elastically, a slight decrease in stress

occurred. From that moment on, the quasi-plateau phase began. Depending on the porosity, a different strain hardening effect was observed. The slope of the compression curves in the quasi-plateau range for the Ti-25%AC and Ti-50%AC specimens was almost the same, which indicated a similar degree of strain hardening, while the strain hardening of the material of the Ti-75%AC specimens was clearly smaller. Nevertheless, it should be noted that the slope of the compression curves in the part corresponding to the quasi-plateau phase was not only the result of the strain hardening of the specimen material but also the result of the increase in the cross-section of the specimen during compression. On the other hand, however, the slope of the curves of the Ti-75%AC samples with relatively high porosities of 57% was very similar in the initial stage of compression, and, after exceeding the strain value of 0.2, it started to increase, especially from the strain of 0.4. This different behavior of the Ti-75%AC material was probably due to the presence of air enclosed in the pores. As stated in [25], the gas effect was significant at the densification stage, wherein the crushing stress, i.e., the strain hardening effect, could be remarkably enhanced. This issue will be further addressed in Section 4.2 on the strain rate sensitivities of tested titanium-based porous materials.

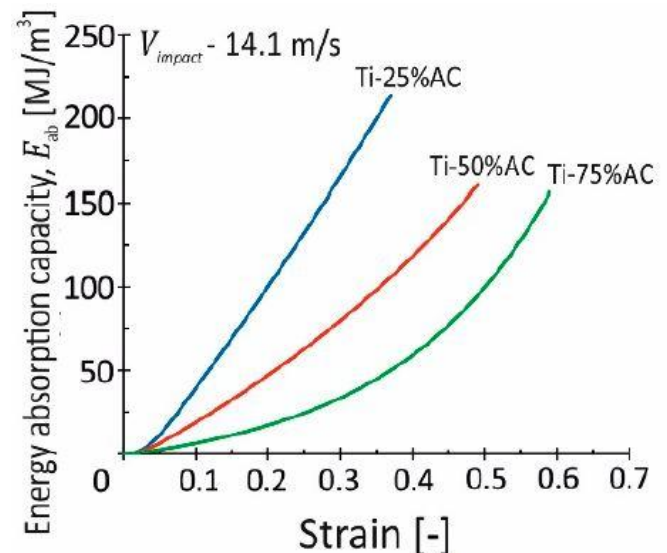


Fig.6 Energy absorption capacity–strain curves of Ti porous materials

The energy absorption capacity–strain curves were calculated according to the dynamic stress–strain curves (Figure 4) in the determined range of densification strain and are shown in Figure 6. The curves of E_{ab} show a linear or parabolic increase with the strain rising. The Ti-25%AC materials reveal a near-linear dependence, while Ti-50%AC and especially Ti-75%AC are characterized by a parabolic relation between the E_{ab} parameter and nominal strain. At the densification strain, the values of E_{ab} are 214, 160, and 156 MJ/m³ for Ti-X%AC, respectively. These results prove that the porosity and fracture mechanisms play a very important role in the energy absorption property. The titanium matrix remains plastic until the porous structure is fully densified.

Conclusions.

The mechanical behavior of the porous titanium materials prepared via powder metallurgy approaches was studied under quasi-static and dynamic compression loading conditions. The influence of porosities, phase composition features, and microstructures on the mechanical behaviors of materials were

analyzed. Based on the experimental data presented and their analysis, the following conclusions could be drawn:

- The mechanical behavior under quasi-static and dynamic compression of porous titanium materials obtained by powder metallurgy method was determined the porosity value. Single-phase porous titanium produced with ammonium carbonate demonstrated a mechanical behavior that was similar to a solid metal if the material porosity was lower than 50%. Otherwise, the tested Ti porous materials behave similar to metal foams. The technological parameters (initial particle size, pressing and sintering conditions) of production were optimized. Sintered materials demonstrate similar shrinkage levels and uniform microstructure within each layer and complete integration between layers of different chemical composition, providing sufficient properties for anti-ballistic applications.

- It was found that porous titanium materials were sensitive to the strain rate when the latter exceeded 103 s^{-1} . The dependence of the strain rate sensitivity on the porosities of specimens was ambiguous, probably due to the effect of the presence of gases trapped in the pores.

- The porous titanium matrix produced using ammonium carbonate demonstrated plastic deformation behavior until the porous structure was fully densified, allowing the accumulation of essentially more mechanical energy, possessing higher energy absorption capacity.

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