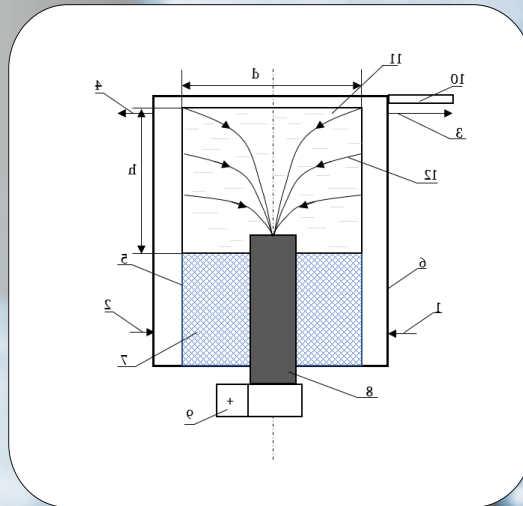


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Furnace for the electroslag remelting of non-metallic household waste

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Abstract: The shortcomings of existing waste-processing furnaces have been analyzed, and a new furnace design for electroslag remelting and combustion of waste, developed at Akaki Tsereteli State University, is proposed. It is demonstrated that its use enables the combustion and remelting of individual waste components without causing environmental pollution from combustion products.

Keywords: waste recycling; electroslag remelting furnace; crystallizer, non-metallic materials, combustion.

1. INTRODUCTION

The processing of household, medical, and other non-metallic industrial waste is a universally recognized problem worldwide. Solving this problem inherently involves resolving two important questions: 1. Determining the scale of the problem solution; 2. Selecting the appropriate technical means, specifically, a furnace suited to the scale of waste thermal processing, or developing a better new one[1].

When determining the scale of the problem, the key factor is the volume of waste to be recycled across the entire country, in two or more regions simultaneously, or in an individual city. It is understood that the construction of factories needed to recycle certain valuable waste must be profitable, since only in this case is it possible to attract investment to address the problem. However, in this case, the population's readiness to sort household waste before disposing of it in bins is also crucial. Otherwise, it is advisable to process household waste at the city or municipal level without sorting, to prevent environmental littering. In parallel, various supportive measures will be needed to cultivate a public demand for waste sorting. In this case, it would be possible to establish a unified waste processing service on a national scale, which would reduce waste processing costs and make it feasible to create enterprises for the beneficial use of waste, for example, factories for processing paper, plastic, and other types of waste, making it profitable while simultaneously solving the problem of environmental pollution from waste.

2. METHODOLOGY

For waste thermal processing, various furnaces can be used based on currently available thermal energy sources. Among these, the most widely used are furnaces powered by liquid fuels, combustible gases, and so-called plasma furnaces.

In furnaces powered by liquid fuels and combustible gases, waste combustion is prolonged; moreover, the gases generated during the process contribute to environmental pollution. The so-called combustion residues are also significant, while the furnaces themselves have a low efficiency coefficient (less than 40%). Plasma furnaces for waste processing have a comparatively higher efficiency coefficient. However, environmental pollution from gases emitted during combustion is significant. Additionally, radiation-related environmental pollution associated with the plasma process is considerable [2]. The protective systems required to mitigate gas and radiation emissions, together with the design of the thermal energy source itself - the plasmatron - complicate plasma furnace construction and contribute to high capital costs. Operating and maintenance costs are likewise high, primarily due to the frequent replacement of plasmatron electrodes.

Within the framework of grant AR-25-2056 funded by the Shota Rustaveli National Science Foundation, we propose a new, original-design furnace for the combustion and remelting of non-metallic materials, specifically household, industrial, and medical waste, known as an electroslag remelting furnace[3]. Figure 1 illustrates the structural diagram of a variant of the so-called crystallizer, which is the section of the furnace where direct waste combustion occurs.

Its design is based on an electroslag remelting furnace for metals, consisting of a power supply, a control system, and a crystallizer. The latter is a key component in which electroslag remelting and metal crystallization occur, enabling the production of an ingot with the desired structure.

The crystallizer is also the main component of the proposed waste-processing furnace, where only melting occurs, without crystallization. However, the old name "crystallizer" has been retained in the presented furnace as well.

When current leads 9 and 10 are connected to the slag bath 11 and the power supply, the bath temperature is maintained by Joule heating resulting from the passage of current 12 through the bath. The magnitude of the temperature depends on the composition of the slag. When the slag contains only feldspar CaF_2 , the temperature of the slag bath is in the vicinity of 900°C . However, with the addition of quicklime CaO , the temperature can reach 1800°C .

3. RESULTS AND DISCUSSION

For creating an experimental waste remelting furnace, it became necessary to calculate the current required for electroslag remelting in order to select a power source for its electrical energy supply.

An analysis of the literature [5] shows that, in addition to the required capacity, one of the main requirements is that the power source should have a steep volt-ampere characteristic, while its open-circuit voltage should not exceed 40 V [6].

If the electrical resistance of the slag in the liquid state is known, then the current required for melting can be determined based on the heat released per unit [of volume/time] as the current passes through the slag bath.

$$Q_1 = 0,24I^2R \text{ kcal}, \quad (1)$$

$$\text{from which } I = \sqrt{\frac{Q}{0,24R}} \quad (2)$$

On the other hand, if the heat capacity C , mass m , and the melting and superheating temperature T_2 of the slag bath are known, taking into account the ambient temperature T_1 , the heat required to melt a mass m of slag can be calculated [7]:

$$Q_2 = cm(T_2 - T_1) \quad (3)$$

Other factors being equal, it is implied that

$$Q_1 = Q_2 = Q \quad (4)$$

Taking into account the height h_s of the slag bath, the inner diameter d of the crystallizer, and the density γ_{sl} of the molten slag, expression (3) takes the form:

$$Q = \pi \frac{d^2}{4} \cdot h_{sl} \cdot \gamma_{sl} \cdot T_m \quad (5)$$

Due to the smallness of the ambient temperature T_1 compared to the melting temperature T_2 of the slag $T_2 \gg T_1$, if we neglect T_1 in expression (3), then the so-called slag-remelting current passing through the slag bath is:

$$I = \sqrt{\frac{\pi d^2 / 4 \cdot h_{sl} \gamma_{sl} C_p T_{\infty}}{0,24 R}} \quad (6)$$

Based on reference material [9], sources on the electroslag remelting of metals, as well as taking into account experimental data [4], the following data were obtained

$$d_{cr} = 25,4 \text{ cm}; \quad h_{sl} = 25,4 \text{ cm} \quad C_{sl} = 1,72 \text{ kJ/kg}; \\ \gamma_{sl} = 2700 \text{ kg/m}^3; \quad T_2 = 1773^\circ\text{K}$$

According to expression (6), the value of the calculated current is $I_{sl} \approx 300 \text{ A}$.

Taking the latter into account, for the power supply of the electroslag remelting installation for non-metallic materials, the constant-current power source VKSM-1000 [7,8] was selected,

which is used for electric arc welding, has a steep volt-ampere characteristic, and an open-circuit voltage of up to 40 V.

In the experiment conducted to evaluate the character of the combustion or slag-melting capability of waste of various compositions, a slag with a composition of 80% CaF₂ + 20% CaO was used, and the temperature of the slag bath, which we measured by means of a pyrometer, was 1200°C. The experimental data are presented in Table 1. The degree of waste combustion was assessed by observing the waste during the combustion process and by recording the combustion process. In the experiment, the criteria for evaluating the quality of the combustion process were: the nature of waste destruction, particularly whether combustion occurs on the surface of the slag or instantaneous melting into the slag occurs, and the amount of combustion products (gases) released as a result of combustion.

The specific gravity of the slag bath in the crystallizer, as well as its viscosity, is directly proportional to the bath temperature[4]. At 1200 °C, the viscosity of the slag is practically equal to that of water. Therefore, the conditions for submerging waste in it are similar to those for submerging waste in water. This is what causes the waste to sink and melt instantly in the slag.

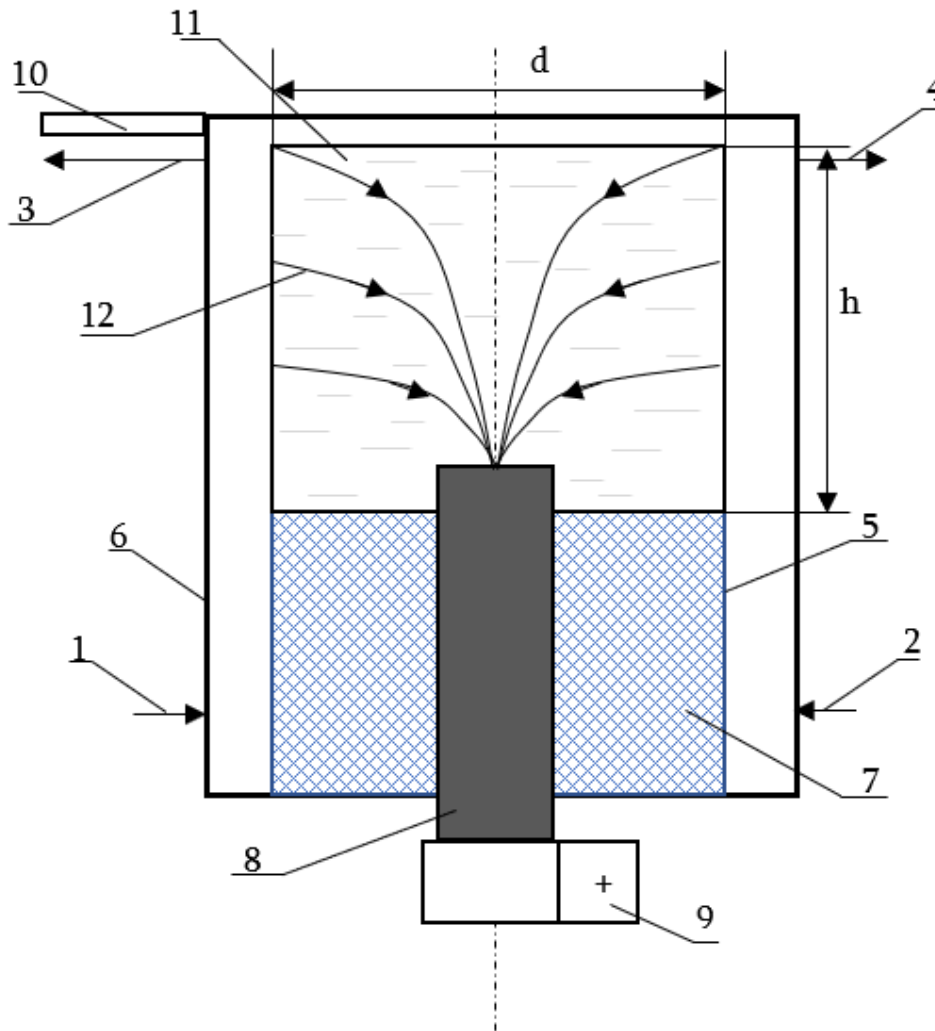


Fig.1. Structural diagram of the so-called crystallizer of the electroslag remelting furnace

1, 2 - water supply points for cooling; 3, 4 - water extraction points for cooling; 5 - inner cylindrical surface of the crystallizer; 6 - outer surface of the crystallizer; 7 - insulating and heat-resistant base; 8 - current lead of the graphite electrode; 10 - current lead to the crystallizer; 11 - molten slag bath; 12 - equivalent lines of current passing through the slag; d - diameter of the crystallizer; h - height of the slag.

According to the above table, paper existing as a separate type of waste is an exception. However, the latter, when mixed with other waste, easily sinks and instantly dissolves in the slag bath. That is, their destruction also occurs practically without residue. The separated residues from the remelting process make a minor but continuous contribution to the increase in slag bath height. They primarily occupy the upper part of the slag bath and gradually overflow out of the crystallizer

By changing the composition of the slag, it became possible, owing to the corresponding change in the resistance of the molten slag, to vary the open-circuit voltage of the power source of the electroslag remelting furnace for non-metallic materials by 40 ± 5 V, which ensured a change in current during the remelting process of up to 300 ± 10 amperes, and, correspondingly, a change in the slag bath temperature within the required range of $1773^\circ\text{K} \pm 100^\circ\text{K}$.

Table 1.: Characteristics of the waste combustion and melting process

No	Type of waste	Characteristics of the waste combustion and melting process	Note
1	Vegetables: potatoes, tomatoes, watermelons, eggplants, melons, cucumbers, beans	Instantly melted, without the release of combustion products	Mainly in small separate portions in the form of shavings
2	Paper	Initially, with combustion, accompanied by the release of a correspondingly small quantity of combustion products, after which it melted. At this point, the release of combustion products ceased completely	Crumpled
3	Plastic		In a compressed state
4	Baby hygiene diaper	Instantly melted, without the release of combustion gases	In a used state
5	Paper mixed with other wastes	Combustion with instantaneous melting, with minimal release of combustion products	With vegetable wastes
6	Plastic mixed with other wastes	Instantaneous melting, with minimal release of combustion products	With vegetable wastes

4. CONCLUSIONS

1. The proposed waste recycling furnace, as can be seen from the characteristics of the remelting process, is more promising than other types of furnaces currently used for the same purpose, including plasma furnaces.

2. In the furnace we proposed, the melting of waste is possible without combustion, which practically eliminates environmental pollution by combustion gases.

3. According to the method for calculating the current required for the electroslag remelting of non-metallic materials, the selected power source of the furnace ensures the required magnitude of the slag bath temperature and the required range of regulation

ACKNOWLEDGMENT

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5. REFERENCES

1. L. Zivzivadze, A. Kakauridze, T. Tskipurishvili, M. Shalamberidze, S. Silagadze. Electroslag Remelting Furnace for Non-Metallic Materials. IX POLAND-GEORGIAN INTERNATIONAL SCIENTIFIC-TECHNICAL CONFERENCE "TRANSPORT BRIDGE EUROPE-ASIA". 2025. Pp. 73-78
2. EUROPEAN PATENT EP0 759 525 A2. <https://patents.google.com/patent/EP0759525A2>
3. Patent GE P 2019 7044 B, გპზო C22B9/187, H05B7/08 [link](https://www.sakpatenti.gov.ge/ka/search_engine/view/14147/1/)
4. S. Silagadze. Study of the electric field in the process of obtaining the turbogenerator rotor blank by electro-slag re-melting, of the Doctoral Thesis Nominated for the Academic Degree of the Doctor of Engineering.
5. Bratkova, O. N. *Power Sources for Arc Welding*. Moscow: Energiya, 1986. 120 p.
6. Medovar, B. I., Shevtsov, V. L., Marinsky, G. S., et al. *Thermal Processes in Electroslag Remelting* / edited by B. I. Medovar. - Kiev: Naukova Dumka, 1978. 308 p.
7. Орлов Б.Д. Технология и Оборудование Электроконтактной точечной сварки. М.:Машиностроение.1986. 800 стр.Orlov, B. D. *Technology and Equipment for Electric-Contact Spot Welding*. Moscow: MASHINOSTROENIE, 1986. 800 p.
8. *Problems of Special Electrometallurgy*. Symposium Proceedings. Kiev: Naukova Dumka, 1979. 327 p.
9. Kutateladze, S. S. *Heat Transfer during Condensation and Boiling*. Moscow: MASHGIZ, 1949. 320 p.

Utilization of powder forging technology for the design of hypercompositional structures from powders of various heat-resistant materials

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Abstract: This study evaluates the applicability of Powder Forging for producing hypercompositional structures from heat-resistant powders based on Inconel 625, ZhS6U, and CoCrAlY alloys. One-step forging and triple forging at 1150 °C were examined to determine their influence on densification, prior particle boundary elimination, microstructural evolution, and high-temperature mechanical behaviour. Triple forging markedly improved interparticle bonding, reduced structural defects, and enabled the formation of dense composite architectures with statistically distributed constituents. The Inconel 625-ZhS6U and Inconel 625-CoCrAlY systems demonstrated high yield strength over a broad temperature range and exhibited anomalous strengthening at elevated temperatures, attributed to dynamic strain aging and phase redistribution. The results confirm that Powder Forging is a promising thermomechanical route for designing advanced heat-resistant powder composites for aerospace hot-section applications.

Keywords: POWDER FORGING, NICKEL-BASED SUPERALLOYS, STRUCTURE, STRENGTH

1. Introduction

Nickel-based superalloys are indispensable materials for key components in modern aerospace engines and industrial gas turbines due to their outstanding creep resistance, high-temperature strength, and excellent oxidation resistance. In aircraft and unmanned aerial vehicles (UAVs), the performance of the power unit is strictly determined by hot section structures, such as turbine blades, disks, and nozzles, which withstand enormous thermal and mechanical loads [1–6]. To maximize thermal efficiency and minimize fuel consumption, turbine designs aim to reduce weight while ensuring the highest possible operating temperatures.

Ni-based alloys, particularly various modifications of Inconel alloys, are widely used in aircraft engines due to their relative affordability and efficiency [6–9]. However, in the cast state, these alloys tend to develop dendritic structures and segregation zones. While conventional casting-deformation technologies are lengthy and costly, alternative approaches like additive manufacturing (AM) still require significant post-processing to eliminate low productivity and structural defects [10–12]. Among powder metallurgy methods, hot pressure processing is gaining traction because it avoids residual porosity [13–17]. Specifically, dynamic hot deformation methods allow the production of highly dense samples with a homogeneous fine-grained structure at relatively low temperatures. Furthermore, powder metallurgy opens up unique possibilities for tailoring material properties through multi-scale structural modifications. On one hand, it allows the design of advanced hybrid materials by blending different superalloy powders (e.g., combining Inconel with high-performance ZhS-type alloys) to optimize the chemical composition and γ/γ' phase balance. On the other hand, this approach facilitates the incorporation of secondary strengthening phases, such as nano-sized oxides or hard carbide particles, into the matrix. Such combined reinforcement effectively pins grain boundaries, suppresses grain growth at ultra-high temperatures, and boosts the overall high-temperature mechanical response beyond the limitations of conventional single-component alloys. [18–20].

As an alternative to AM technology, the Frantsevich Institute for Problems of Materials Science (NAS of Ukraine) has been developing a powder technology known as Direct Powder Forging (DPF) [21–25]. Within this method, the desired product shape is formed during the cold and hot deformation of powders, and the required final structure is achieved by optimal thermomechanical treatment (TMT). Generally, direct powder forging (DPF) techniques are highly effective for grain refinement and controlling phase precipitation, suppressing grain growth, and promoting the uniform distribution of strengthening phases (such as γ or γ'' precipitates). However, implementing a multiple DPF method on highly alloyed, heat-resistant superalloys often introduces complex deformation mechanisms, including localized shear banding, mechanical twinning, and dynamic recrystallization. The exact

correlation between triple forging parameters and structural homogeneity is still a subject of ongoing debate.

To address this gap, is to model the distribution of flow deformation characteristics and porosity changes during hot powder forging, alongside developing thermomechanical processing regimes for the Ni-based superalloy powder alloy. This study investigates the microstructural evolution and material behaviour at each forging stage to achieve an optimal set of mechanical properties over a wide temperature range, providing practical guidelines for optimizing industrial forging processes of advanced superalloys.

2. Materials and experimental procedure

Spherical powders of the nickel-based superalloys Inconel 625 and ZhS6U, produced by the plasma rotating electrode process (PREP), as well as powder of the cobalt-based heat-resistant alloy Co-22.3Cr-10.8Al-0.35Y (wt.%), were used in the study. The densification and deformation behavior of the Inconel 625 and ZhS6U powder alloys, as well as powder mixtures of Inconel 625 – 50 vol.% ZhS6U and Inconel 625 – 50 vol.% Co-22.3Cr-10.8Al-0.35Y, were investigated. The superalloy powders were blended in a 3D mixer for 3 h. The required amounts of the heat-resistant alloy powders were loaded into a cylindrical steel container, followed by hermetic compaction at a pressure of 450 MPa (Fig. 1).

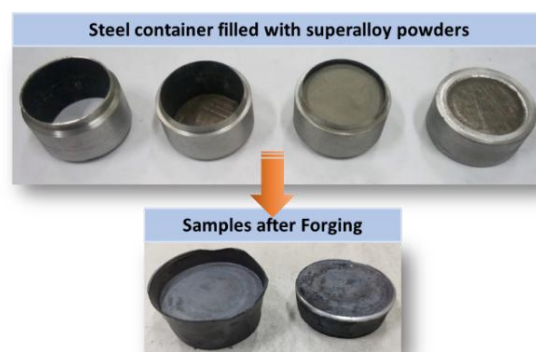


Fig. 1. Steel container with superalloy powders before and after processing by the DPF scheme.

The prepared powder-filled containers were subsequently forged using an FB1732 screw press with a nominal force of 1.6 MN and a kinetic energy of the moving parts at the end of the acceleration stroke of at least 6.18 kJ. The punch speed during compaction was 0.35 m/min.

Forging was performed in a semi-closed die with an upper flash. For the Inconel 625 and ZhS6U alloys, the densification and deformation behaviour of the powder compacts were investigated under single-pass and three-pass forging at 1150 °C. Based on the deformation data obtained for Inconel 625 and ZhS6U, thermomechanical processing of the Inconel 625 – 50 vol.% ZhS6U and Inconel 625 – 50 vol.% Co-22.3Cr-10.8Al-0.35Y powder mixtures was subsequently investigated at 1150 °C using a three-pass forging schedule (Fig.2).

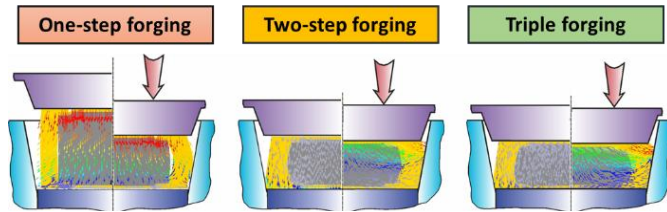


Fig. 2. Schematic of multiple forging of a heat-resistant alloy using the DPF method.

The specimens were heated by immersing them in a furnace preheated to the required temperature under an argon atmosphere and holding them at this temperature for 20 min. After forging, conical specimens with a flash diameter of 42 mm and a height of 9 mm were obtained (Fig.1). For the multiple-forging procedure, the conical specimen obtained after the first deformation step was machined to a cylindrical shape, rotated by 180°, and subjected to the second deformation step until a height of 11 mm was reached. The third and final deformation step was performed according to the same procedure. The material structure was examined using a JEOL Superprobe-733 and Tescan Vega 3 scanning electron microscopes. Compressive strength was measured on samples of 5×3×3 mm at temperatures of 20 and 750 °C using a UTM-100 universal testing machine.

3. Results and discussion

The DPF technology involves structural evolution through the localization of deformation in the region of interparticle boundaries. As a result, two important structural transformations are achieved: (I) recrystallization of interparticle boundaries in the contact regions and (II) the formation of an ultrafine-grained structure through the dynamic recrystallization (DRX) mechanism. Previous studies [24, 25] demonstrated that DRX indeed initiates in the boundary regions during a single forging operation; however, relatively high residual porosity and crack-like defects remain at the triple junctions of particles. To eliminate these structural defects, an advanced DPF technology involving three consecutive forging operations was proposed. Microstructural investigations and mechanical testing showed that interparticle fracture was eliminated after triple forging, although the yield strength decreased slightly due to grain growth. This technology also enables the fabrication of complex powder composites from different types of starting powders.

Fig. 3 presents the results of mechanical testing of ZhS6U and Inconel 625 alloys after forging at 1150 °C using one and three deformation steps. As expected, the yield strength of both alloys was higher after a one - step forging operation than after triple forging.

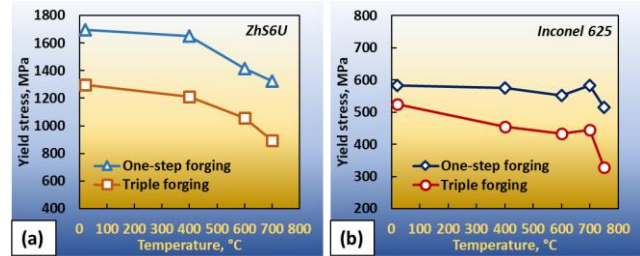


Fig. 3. Yield stress versus temperature for (a) ZhS6U and (b) Inconel 625 after different forging conditions.

Microstructural analysis shows that a large number of planar defects remain after one-step forging (Fig. 4a, 5a). Agglomerates of impurities, carbide particles, and γ' precipitates are predominantly concentrated along the prior particle boundaries (PPBs), as shown in Fig. 4a. The fracture mode for both ZhS6U and Inconel 625 is interparticle (Figs. 4b, 5b). A distinctive feature of the processing of the ZhS6U alloy is that deformation is performed below the solvus temperature, and the strengthening γ' precipitates remain within the matrix. In contrast, Inconel 625 is processed above the solvus temperature, at which the strengthening γ'' precipitates dissolve into the matrix, while the carbide particles remain. After triple forging, an increase in grain size is observed; however, the interparticle boundaries are almost completely eliminated (Figs. 4c, 5c). Repeated deformation breaks up the initial interparticle boundaries and promotes a more uniform distribution of second-phase particles throughout the matrix, although small localized clusters and some larger individual particles still remain. The fracture mode changes to transcrystalline fracture (Figs. 4d, 5d).

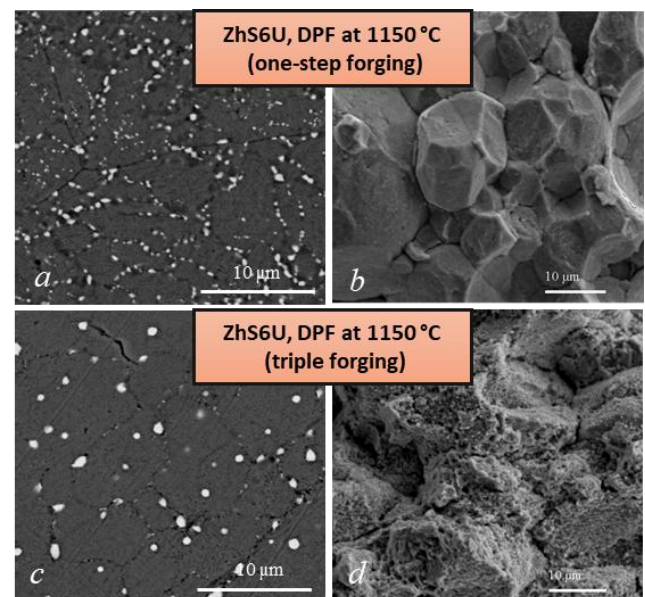


Fig. 4. Microstructure (a, c) and fracture surface (b, d) of ZhS6U powder alloy after deformation processing at a temperature of 1150°C.

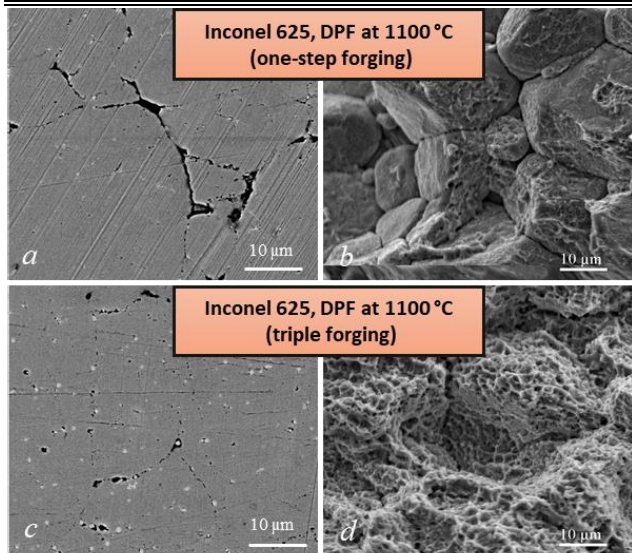


Fig. 5. Microstructure (a, c) and fracture surface (b, d) of Inconel 625 powder alloy after deformation processing at a temperature of 1150°C.

For the alloy consisting of 50% ZhS6U and 50% Inconel 625, the mechanical testing results (Fig. 6a) after triple forging demonstrated a room-temperature yield strength comparable to that of the stronger ZhS6U alloy, reaching approximately 1000 MPa. With increasing temperature to 500 °C, the yield strength decreased to approximately 700 MPa, which may be associated with the softening effects characteristic of Inconel 625. However, at higher temperatures, an anomalous temperature dependence of the yield strength was observed, with the yield strength increasing to approximately 950 MPa at 750 °C. This behaviour may be related to the dynamic strain aging (DSA) mechanism, which is characteristic of Inconel 625 in this temperature range.

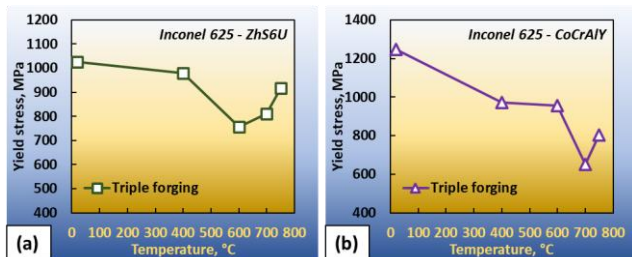


Fig. 6. Yield stress versus temperature for (a) Inconel 625-ZhS6U and (b) Inconel 625 - CoCrAlY powder alloy after different forging conditions.

No evidence of planar defects is observed at the boundaries between the Inconel 625 and ZhS6U powder particles (Fig. 7). Considering the high deformation rate of 10^2 s^{-1} , the structure does not have sufficient time to become homogenized through diffusion. The resulting alloy can be considered a hyper-composite with a statistically distributed structure and sufficient connectivity of each structural constituent. Under optimized deformation conditions, this approach enables the structure and properties to be tailored by exploiting the beneficial contributions of the individual structural constituents.

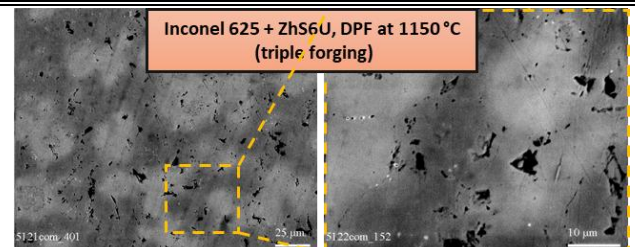


Fig. 7. Structure of Inconel 625-ZhS6U powder alloy after deformation processing at temperature 1150°C (general view and high-magnification image showing grain boundaries).

Figure 6b presents the results of mechanical testing of the Inconel 625 - 50 vol.% Co-22.3Cr-10.8Al-0.35Y powder composite. The mechanical properties are also comparable to those of the best high-temperature superalloys. At room temperature, the yield strength reaches 1200 MPa and remains at approximately 1000 MPa up to 600 °C, followed by a decrease to 650 MPa at 700 °C. However, the yield strength increases again to 800 MPa at 750 °C (Fig. 6b). This anomalous temperature dependence of the yield strength can be attributed to the characteristics of the dynamic strain aging (DSA) effect in this temperature range.

Microstructural analysis shows that, in addition to the two structural constituents characteristic of each component of the composite, a relatively wide transition zone forms between the dissimilar powder particles as a result of the diffusion of alloying elements into the interparticle contact regions (Fig. 8a). Although no crack-like defects are observed in the microstructure, the fracture path reflects the structure of the initial powder particles, apparently due to the higher brittleness of the β -phase (CoAl) formed in the interparticle region (Fig. 8b).

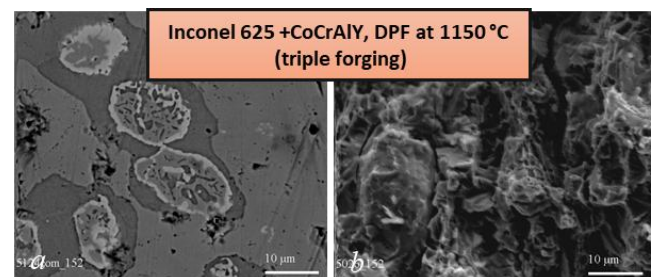


Fig. 8. Microstructure (a) and fracture surface (b) of Inconel 625-CoCrAlY powder alloy after deformation processing at a temperature of 1150°C.

Thus, DPF technology enables the development of advanced heat-resistant materials by designing hyper-composites comprising constituents of different heat-resistant powder alloys.

4. Conclusions

Implementation of multiple Direct Powder Forging (DPF) at 1150 °C successfully ensures enhanced consolidation, structural homogenization, and tailored mechanical performance in advanced powder-derived nickel-based systems. Microstructural analysis confirms that shifting from single-step to three-pass forging effectively disrupts prior particle boundaries (PPBs) and accelerates pore closure, yielding highly dense matrices with well-bonded interfaces for both the individual alloys (ZhS6U, Inconel 625) and their

blended mixtures (Inconel 625 – ZhS6U/CoCrAlY). Mechanical evaluations over a wide temperature range reveal that triple forging consistently induces a softening effect (through coarsening of the microstructure and redistribution of the strengthening phase), compared to one-step forging, resulting in increased material ductility. Furthermore, the powder composites after triple forging demonstrate distinct anomalous high-temperature strengthening peaks between 600 °C and 750°C driven by dynamic aging and phase redistribution. These findings provide critical guidelines for optimizing multi-directional hot deformation regimes to fabricate highly efficient, heat-resistant components for aerospace structures. DPF technology expands the possibilities for developing heat-resistant materials through the design of hyper-composites comprising constituents of different heat-resistant powder alloys.

5. Acknowledgements

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6. References

1. Gudivada, G., & Pandey, A. K. (2023). Recent developments in nickel-based superalloys for gas turbine applications. *Journal of Alloys and Compounds*, 963, 171128.
2. Çoban, S., & Oktay, T. (2018). Unmanned aerial vehicles (UAVs) according to engine type. *Journal of aviation*, 2(2), 177-184.
3. Dutczak, J. (2016, September). Micro turbine engines for drones propulsion. In IOP Conference Series: Materials Science and Engineering (Vol. 148, No. 1, p. 012063). IOP Publishing.
4. Large, J., & Pesyridis, A. (2019). Investigation of micro gas turbine systems for high speed long loiter tactical unmanned air systems. *Aerospace*, 6(5), 55.
5. Pollock, T. M., & Tin, S. (2006). Nickel-based superalloys for advanced turbine engines: chemistry, microstructure and properties. *Journal of propulsion and power*, 22(2), 361-374.
6. Morad, A. M., & Shash, Y. M. (2014, May). Nickel base superalloys used for aero engine turbine blades. In The International Conference on Applied Mechanics and Mechanical Engineering (Vol. 16, No. 16th International Conference on Applied Mechanics and Mechanical Engineering., pp. 1-22). Military Technical College.
7. Mittendorf, D. (2014). U.S. Patent No. 8,858,873. Washington, DC: U.S. Patent and Trademark Office.
8. Cunningham, C. S., Ransom, D., Wilkes, J., Bishop, J., White, B. (2015, June). Mechanical design features of a small gas turbine for power generation in unmanned aerial vehicles. In Turbo Expo: Power for Land, Sea, and Air (Vol. 56796, p. V008T23A021). American Society of Mechanical Engineers.
9. Kommel, L. (2017). Microstructure and properties characterization of polycrystalline Ni-Fe-Cr-based superalloy EP-718E after electric upsetting. *Key Engineering Materials*, 721, 467-472.
10. Waugh, I., Moore, E., Greig, A., Macfarlane, J., & Dick-Cleland, W. (2021, March). Additive manufacture of rocket engine combustion chambers using the Abd R-900Am nickel superalloy. In SP2020 Virtual Conf (pp. 17-19).
11. Huang, T. X., Liu, Z., Jiang, D., Ye, S., Liu, T., Zhou, R., & Zhang, Y. (2026). Laser beam powder bed fusion nickel-based superalloys: microstructure, alloy tailoring and mechanical properties. *Virtual and Physical Prototyping*, 21(1), e2619387.
12. Ojo, O. O., & Taban, E. (2023). Post-processing treatments–microstructure–performance interrelationship of metal additive manufactured aerospace alloys: a review. *Materials science and technology*, 39(1), 1-41.
13. Brooks, J. W. (2000). Forging of superalloys. *Materials & design*, 21(4), 297-303.
14. Kienl, C. (2019). Hot forging of the nickel-base superalloy ATI 718Plus (Doctoral dissertation).
15. Laptev, A. V., Tolochin, A. I., Kovalchenko, M. S., Evich, Y. I., & Okun', I. Y. (2016). Structure and properties of Ni3Al intermetallic under vacuum impact sintering. *Powder Metallurgy and Metal Ceramics*, 54(9), 554-567.
16. Park, N. K., Kim, I. S., Na, Y. S., & Yeom, J. T. (2001). Hot forging of a nickel-base superalloy. *Journal of Materials Processing Technology*, 111(1-3), 98-102.
17. Tolochyna, O., Tolochyn, O., Bagliuk, G., Podrezov, Y., Zgalat-Lozynskyy, O., & Okun, I. (2023). Effect of Heating Rate and Hot Forging Temperature on Phase Formation and Complex Physical and Mechanical Properties of Powdered Iron Aluminide: Tolochyna, Tolochyn, Bagliuk, Podrezov, Zgalat-Lozynskyy, and Okun. *JOM*, 75(3), 825-836.
18. Lee, D. W., Kim, J. W., Lee, S. G., Cong, D. V., Kim, J. C., Kim, H. J., ... & Yoon, T. S. (2024). Microstructural characteristics of Modified ods Ni-Based superalloys fabricated using laser-powder Bed fusion. *Archives of Metallurgy and Materials*, 69(2), 467-470.
19. He, W., Liu, F., Tan, L., Huang, L., He, S., & Fan, C. (2024). Flow behaviors and microstructural evolutions of nickel-based ODS superalloys during hot deformation. *Journal of Materials Science & Technology*, 200, 13-26.
20. He, W., Liu, F., Tan, L., Huang, L., He, S., Zhang, L., & Fan, C. (2024). Investigation on different additions as candidates for nano-oxide particles in nickel-based ODS superalloys. *Materials Characterization*, 209, 113648.
21. Tolochyn, O. I., Tolochyna, O. V., Podrezov, Y. M., Danylenko, V. I., & Bagliuk, G. A. (2025). The Role of Deformation Conditions and Mo-Doping on the Evolution of Structure and Mechanical Properties of Powdered Iron Aluminide. *JOM*, 1-14.
22. Tolochyn, O., Tolochyna, O., Bagliuk, G., Yevych, Y., Danylenko, V., & Podrezov, Y. (2024). Mechanical behavior of powdered iron aluminide Fe–28Al manufactured by direct powder forging. *Intermetallics*, 175, 108537.
23. Tolochyn, O., Kyrlyuk, S., Bagliuk, G., Podrezov, Y., & Tolochyna, O. (2025). Multilevel analysis of deformation and structure formation processes in powdered iron aluminide products obtained by different technological schemes of direct powder forging. *International Journal of Material Forming*, 18(1), 1-14.
24. Tolochyn, O., Tolochyna, O., Bagliuk, G., & Podrezov, Y. (2026). Microstructure and Properties of Boride- Strengthened Titanium- Alloyed Iron Aluminides Processed by Direct Powder Forging Under Dynamic Recrystallization. *Advanced Engineering Materials*, e202503141.
25. Tolochyn, O. I., Tolochyna, O. V., Bagliuk, G. A., Kyrlyuk, S. F., Danylenko, M. I., Okun, I. Y., & Podrezov, Y. M. (2026). Effect of Structural Refinement on the Mechanical Properties of Fe–28 Al Intermetallic Alloy Produced by Direct Powder Forging III. Development of High-temperature Consolidation Route for Fe–28 Al Iron Aluminide for Dynamic Recrystallization Conditions. *Powder Metallurgy and Metal Ceramics*, 1-19.

Effect of density gradient on energy absorption properties of additively manufactured tpms-d infill pattern via material extrusion method

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Abstract:

As additive manufacturing (AM) technologies continue to gain traction across various industries, researchers have increasingly focused on optimizing the mechanical performance of polymer parts produced through material extrusion (MEX). Among the manufacturing parameters involved, infill pattern and density stand out as key factors that directly determine the mechanical behavior and energy absorption capacity of printed components. This study examines how different infill density gradients influence energy absorption, focusing specifically on samples built from polylactic acid (PLA) filament using a Triply Periodic Minimal Surface (TPMS-D) diamond pattern. TPMS-based infill patterns were selected for this study due to their potential to offer superior mechanical properties and energy absorption capacity compared to conventional two-dimensional infill patterns. The samples were subjected to quasi-static compression tests, from which stress-strain curves were obtained and energy absorption properties were calculated. The findings of this study are expected to inform the optimization of infill pattern and density parameters for energy-absorption applications, while also offering practical guidance for designing lightweight structures capable of withstanding high-energy impacts.

KEYWORDS: ADDITIVE MANUFACTURING VIA MATERIAL EXTRUSION, INFILL PATTERN, INFILL DENSITY GRADIENT, ENERGY ABSORPTION, TPMS PATTERN

1. Introduction

Triply periodic minimal surfaces (TPMS) are mathematically defined structures characterized by zero mean curvature at every point, repeating periodically along three Cartesian axes. Their smooth, non-self-intersecting topology enables systematic control over relative density and enhances load transfer efficiency. Among TPMS architectures, the Diamond (TPMS-D) structure has demonstrated superior load-carrying capacity and specific energy absorption compared to conventional lattice counterparts [1–5].

Material extrusion (MEX), also known as fused filament fabrication (FFF), is widely employed for fabricating TPMS structures due to its capability to produce complex geometries layer-by-layer. Recent studies have shown that TPMS structures fabricated via this method exhibit excellent compression performance and energy absorption capacity [3,4,6,7].

Introducing density gradients into TPMS architecture represents a promising strategy for enhancing mechanical performance. Gradient TPMS structures have been reported to exhibit superior energy absorption compared to homogeneous counterparts, particularly when the gradient direction aligns with the loading direction, as layer-by-layer deformation enhances energy dissipation [3,7].

Despite growing interest, the specific influence of density gradients on the energy absorption properties of TPMS-D structures fabricated via material extrusion remains insufficiently explored. This study aims to address this gap by systematically investigating the effect of density gradient configurations on the compressive and energy absorption behavior of additively manufactured TPMS-D infill patterns.

2. Material and Method

TPMS-Diamond infilled cubic specimens (25×25×25mm) were produced additively using a Creality K1 Max branded 3D printer, which works with the material extrusion method (MeX). Printing parameters were given in Table 1. Each sample coded using the

start and end infill density settings of the slicer gradient along the loading Z-axis (Table 2). Creality Print 7.2 was used for slicing.

Table 1. Printing parameters

Nozzle diameter	Layer height	Nozzle temperature	Bed plate temperature	Print speed
0.4mm	0,20mm	220 C	50 C	90 mm/min

Table 2. Nomenclature of samples

Sample code	Infill gradient	Infill setting (start → end)	Mean weighed mass (g)
1515	Uniform	15 → 15	3.07
2020	Uniform	20 → 20	4.48
2525	Uniform	25 → 25	4.67
3030	Uniform	30 → 30	4.75
4040	Uniform	40 → 40	5.08
8080	Uniform	80 → 80	6.64
9090	Uniform	90 → 90	7.25
2580	Graded	25 → 80	4.75
1590	Graded	15 → 90	4.72

Table 3. Organization of the nine configurations into four comparison sets

Set	Purpose	Configurations compared
I	Effect of gradient severity at constant mass	3030 (uniform), 2580 (graded), 1590 (graded)
II	Graded architecture versus the uniform counterparts of its gradient endpoints	1515, 1590, 9090
III	As Set II, for a milder gradient	2525, 2580, 8080
IV	Uniform architectures bracketing the Set I baseline	2020, 3030, 4040

Two layers of bottom and top layers were printed for each sample to obtain planar loading surfaces. After production, the samples were weighed using a precision balance to be used in calculating the specific mechanical property data. Then, compression tests were carried out on a UVE MNR100 testing machine with a crosshead speed of 20 mm/min. Using the data obtained from compression tests mechanical properties were calculated to evaluate the effect of density parameter. The purpose of the comparison sets with sample codes were given in Table 3.

3. Result and Discussion

3.1 Set I - Effect of gradient severity at constant mass (3030, 2580, 1590)

The aim of the set is to separate the effect of grading from the amount of material. Since the masses are approximately the same, all differences are due solely to the gradient. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure1 and 2.

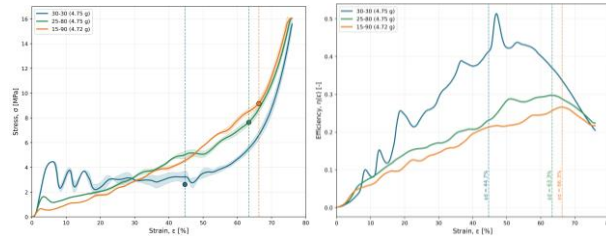


Fig. 1. Compressive response of the equal-mass configurations 30-30, 25-80 and 15-90 (Set I) a) Stress-strain graph, b) Energy absorption efficiency

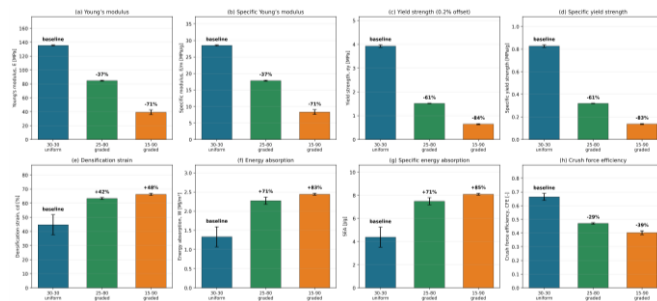


Fig. 2. Mechanical properties of Set I a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Densification strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

Although grading significantly improved energy absorption, it caused deterioration in Young's modulus and yield strength. A moderate gradient reduced the elastic modulus by 37.4% and yield strength by 61.4%, while increasing SEA by 70.8%; a severe gradient reduced the elastic modulus by 71.1% and yield strength by 83.6%, while increasing SEA by 84.6%.

Similar findings regarding the compressive properties of lattice structures were also obtained by Lin et al and Distefano et al [4,8]. Interestingly, in their study, Silva et al. concluded that the use of cell thickness gradients can promote structures with higher rigidity and absorbed energy that can compete with traditional sandwich panel core structures [9].

Increasing the density transition from 2580 to 1590 resulted in a stiffness loss of 33.7 MPa while providing only a 13.8 J/g SEA gain. Therefore, 2580 emerged as a more efficient option in load-bearing applications.

When energy absorption behavior is examined regionally, it is seen that at low strain values, the uniform structure absorbs much more energy per unit mass, but the graded structures take the lead at approximately 50% strain. Therefore, concluding that "grading absorbs better" becomes meaningless without specifying the usable crushing distance.

The SEA scattering decreases from 19.7% to 4.1% and 1.0%. In the uniform structure, the collapse band starts where the worst defect is located, while in the graded structure it always occurs in

the weakest layer. This indicates that the uncertainty in deformation behavior is eliminated with grading.

3.2 Set II - The severe gradient against the uniform counterparts of its endpoints (1515, 1590, 9090)

The aim of the set was to test whether the 1590 gradient provided an intermediate value between uniform 1515 and uniform 9090. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure 3 and 4.

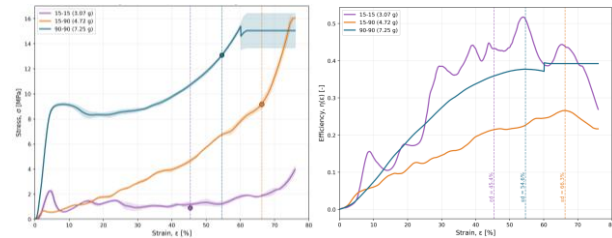


Fig. 3. Compressive response of the graded 15-90 architecture and the uniform architectures of its gradient endpoints, 15-15 and 90-90 (Set II) a) Stress-strain graph, b) Energy absorption efficiency

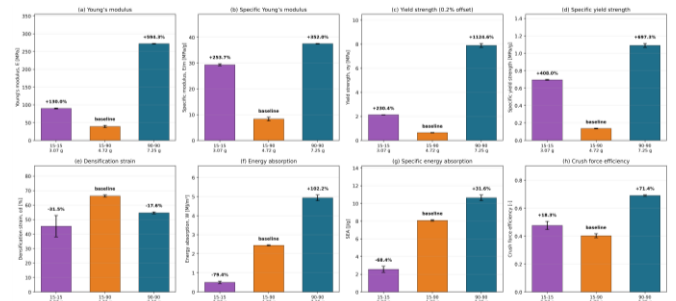


Fig. 4. Mechanical properties of Set II a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Densification strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

The Young's modulus of the 1590 sample was obtained as 39.2 MPa, while the Young's modulus of uniform 1515 was 90.1 MPa. The uniform 1515 sample had 130% higher stiffness and 230% higher yield strength compared to the graded sample, while the graded sample was 54% heavier. This led to even greater increases in specific mechanical properties. However, if the structure in the weakest layers of these two samples is the same, similar behavior, especially in the elastic region, would be expected. This indicates that the mechanical behavior cannot be explained by the logic of series springs. Although analyses to determine the cause of this behavior – such as micro-CT – were not performed within the scope of this study, the possibilities of inadequate interlayer adhesion in the density transition and the rupture of the bond between cells can be evaluated.

Another finding is that the strong gradient eliminates the initial peak. While both uniform structures show distinct initial peaks, this behavior disappears in the graded sample. This indicates that gradient designs can be preferred depending on the need, since the peak force determines the acceleration transmitted to the body.

Unlike other uniform structures, the graded sample exhibited a monotonically increasing stress-strain curve; this resulted in a higher densification point.

If there is no mass constraint, it can be concluded that this behavior improves with increasing mass in terms of energy absorption capacity. This was observed in both energy absorption and specific energy absorption values.

3.3 Set III - The moderate gradient against the uniform counterparts of its endpoints (2525, 2580, 8080)

The configurations examined under Set III were evaluated to validate the severe gradient effects observed in Set II under a more moderate gradient profile. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure 5 and 6.

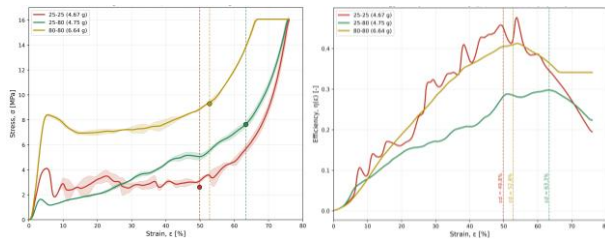


Fig. 5. Compressive response of the graded 25-80 architecture and the uniform architectures of its gradient endpoints, 25-25 and 80-80 (Set III) a) Stress-strain graph, b) Energy absorption efficiency

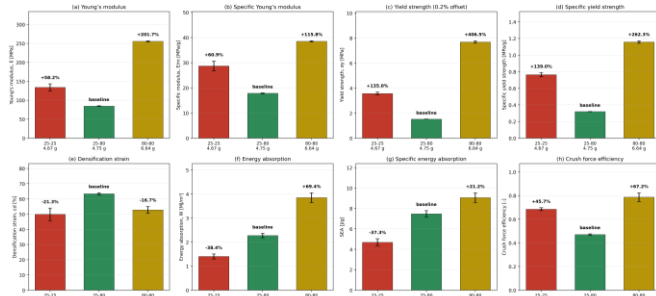


Fig. 6. Mechanical properties of Set III a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Denatification strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

The tendency for stiffness and strength loss was also maintained in the moderate gradient series. The 8080 configuration exhibited a 201.7% decrease in elastic modulus and a 406.5% decrease in yield strength compared to the 2580 gradient. It was determined that the stiffness difference loss increased as the gradient width increased. This consistent trend obtained in two independent sample sets and at different mass levels indicates that the performance loss is systematic behavior [8].

Unlike the severe gradient structures, it was found that the moderate gradient profile maintained a distinct stress peak. When evaluated together with the Set I results, increasing the gradient amplitude does not eliminate the stress peak alone, but dampens it above a certain critical threshold. This indicates the existence of a threshold value between mechanisms for peak mitigation and complete elimination.

The uniform 8080 configuration yielded the most stable mechanical response throughout the experimental study. The structure exhibited a Crush Efficiency (CFE) of 0.787 and a plateau stress dispersion of only 9.2%. Within the evaluated strain range, it was observed that the thick cell walls deformed via a plastic deformation mechanism rather than brittle buckling.

3.4 Set IV — Uniform architectures around the Set I baseline (2020, 3030, 4040)

Set IV (2020, 3030, and 4040) was investigated to verify the representativeness and boundary conditions of the 3030 cellular configuration, which was considered the basic reference in Set I analyses. Obtained compressive response graphs and calculated mechanical properties of the Set are shown in Figure 7 and 8.

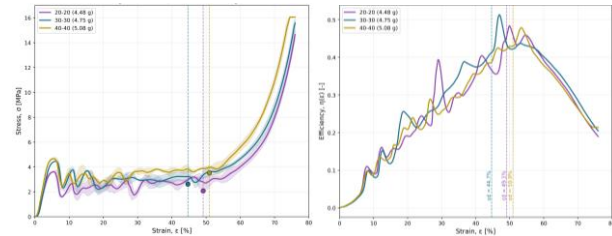


Fig. 7. Compressive response of the uniform architectures 20-20, 30-30 and 40-40 (Set IV) a) Stress-strain graph, b) Energy absorption efficiency

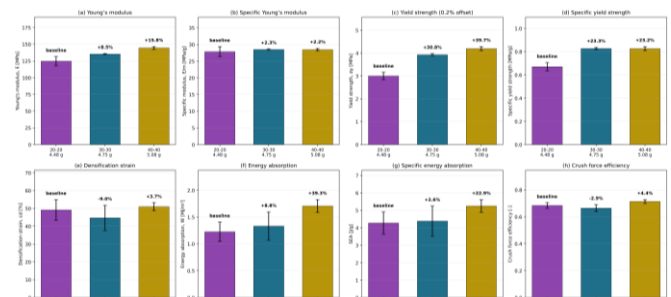


Fig. 8. Mechanical properties of Set IV a) Young's modulus, b) Specific Young's modulus, c) Yield strength, d) Specific yield strength, e) Denatification strain, f) Energy absorption, g) Specific energy absorption, h) Crush force efficiency

It was determined that the 3030 configuration exhibits a monotonic transition between neighboring configurations (2020 and 4040) in terms of elastic modulus, yield strength, and specific modulus values. This confirms that the relationships observed in Set I are not based on a biased reference point. However, the 3030 structure remained somewhat lower than the other samples in terms of densification strain.

In the mass range of 4.48 g to 5.08 g (transition from 2020 to 3030), specific stiffness and specific yield strength showed an increase of 2.3% and 23.3%, respectively. However, in the range of 5.08 g to 7.25 g (from 3030 to 4040), the specific modulus and specific yield strength values remained constant within certain limits, forming a plateau. This indicates saturation in specific mechanical parameters at a certain value and shows that the added material does not contribute to the specific mass efficiency.

It was determined that the configuration with the highest density does not always offer the most efficient mechanical behavior. Plateau uniformity reached the optimum level at 6.64 g mass with 9.2%, then decreased to 13.9% at 7.25 g mass; Simultaneously, the CFE value also decreased from 0.787 to 0.690. This performance loss was accompanied by an increase in the ratio of stress to plateau stress in the densification region from 1.20 to 1.33. It is assessed that the low internal void volume in the high-density configuration compresses the cell wall contacts into a narrower strain range, leading to early densification hardening.

4. Conclusion

In this study, the mechanical response and energy absorption characteristics of additively manufactured PLA components with uniform and functionally graded TPMS-Diamond infill patterns were systematically investigated through quasi-static compression tests. Based on a four-set comparative experimental framework, the principal findings are summarized as follows:

- At constant mass, density grading significantly enhances energy absorption capacity at the expense of elastic modulus and yield strength. A moderate gradient (2580) increased specific energy absorption (SEA) by 70.8%, whereas a severe gradient (1590) yielded an 84.6% increase in SEA compared to uniform counterparts. However, uniform architectures consistently exhibited higher load-bearing capacity and stiffness than graded structures of equivalent average relative density.
- Suppressing the initial stress peak requires exceeding a critical gradient severity threshold. While the moderate gradient (2580) dampened the peak stress by 60.2% (retaining a minor overshoot at $\epsilon \approx 3.4$, the severe gradient (1590) eliminated the stress peak, yielding a monotonic, stable plateau. Furthermore, grading transformed the stochastic collapse typically observed in uniform lattices into a deterministic, layer-by-layer progressive deformation, dramatically reducing the coefficient of variation in SEA from 19.7% to 1.0%.
- The elastic modulus of graded architectures unexpectedly fell below that of the uniform architecture representing their lowest gradient endpoint (by 56.5% for 1590 and 36.8% for 2580). This reproduced phenomenon indicates that global stiffness in graded MEX structures cannot be modeled simply as springs in series, likely due to localized stress concentrations and inter-cell boundary compliance in low-density transition layers.
- Mass efficiency within uniform TPMS-D structures exhibits non-monotonic behavior. Specific stiffness and specific strength saturate in the medium-density range (4.48–5.08 g), whereas maximum structural performance, plateau stability, and crush force efficiency (CFE = 0.787) peak at 6.64 g (80–80 setting). Beyond this point (90–90 setting), early cell-wall contact accelerates densification hardening and degrades CFE to 0.690.

Future work should prioritize examination of the gradient transition zones and the introduction of intermediate gradient severities to resolve the peak-suppression threshold.

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5. References

- [1] Abdulazizov M and Pervaiz S 2025 Comprehensive Analysis of the Compressive Behavior of Fischer-Koch-S, F-Rd, and Neovius TPMS Cellular Structures in 3d-Printed PLA Specimens *Journal of Elastomers & Plastics*
- [2] Ahmad A, Belluomo L, Bici M and Campana F 2023 Bird's Eye View on Lattice Structures: Design Issues and Applications for Best Practices in Mechanical Design *Metals* **13** 1666
- [3] Hossain M S, Hossain Md M and Nilufar S 2025 An Overview of Additive Manufacturing of Triply Periodic Minimal Surface (TPMS) Structures *Polymers* **17** 3307
- [4] Qin D, Sang L, Lai S and Zhao Y 2022 Compression Performance and Deformation Behavior of 3d-Printed PLA-Based Lattice Structures *Polymers* **14** 1062
- [5] Weber D and Srinivas Sundarram S 2023 3D - printed and foamed triply periodic minimal surface lattice structures for energy absorption applications *Polymer Engineering & Sci* **63** 1133–45
- [6] Öztürk F H, Temiz A and Pehlivan F 2026 Numerical and Experimental Investigation of Failure Mechanisms in Additively Manufactured Multimaterial Triply Periodic Minimal Surface Polymer Cellular Materials *Advanced Engineering Materials* **28**
- [7] Sankineni R and Ravi Kumar Y 2022 Evaluation of energy absorption capabilities and mechanical properties in FDM printed PLA TPMS structures *Proceedings of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science* **236** 3558–77
- [8] Distefano F and Epasto G 2024 Effect of density grading on the mechanical behaviour of advanced functionally graded lattice structures *Journal of the Mechanical Behavior of Biomedical Materials* **153** 106477
- [9] Garrido Silva B, Alves F, Sardinha M, Reis L, Leite M, Deus A M and Vaz M F 2022 Functionally graded cellular cores of sandwich panels fabricated by additive manufacturing *Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications* **236** 1814–28

Precision determination of metal content in neutron super-mirrors by X-ray fluorescence method

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Abstract: Neutron polarizing super-mirrors are aperiodic multilayer structures made of alternating magnetic CoFe and non-magnetic TiZr layers deposited on glass or silicon substrates using the magnetron sputtering method. The polarizing ability of such super-mirrors and the efficiency of their use depend on the fine ratio of the thickness of the layers and the relative metal content in the layers [1, 2]. X-ray fluorescence analysis (XRF) is highly sensitive, but it has a rather large relative error, usually not less than 5%. An original X-ray spectrometer with a radioisotope source based on ¹⁰⁹Cd was used to determine the content of Fe, Co, Ti, and Zr in super-mirror samples with sufficient accuracy to control their manufacturing technology. The XRF analysis was based on the direct external standard method taking into account absorption corrections. As a result, the relative metal content in the super-mirror layers was determined with a relative error of less than 1%.

KEYWORDS: NEUTRON SUPER-MIRRORS, X-RAY FLUORESCENCE ANALYSIS, X-RAY SPECTRA OF Ti, Mn, Fe, Co, Sr, Zr

1. Introduction

Neutron super-mirrors are the basic components of neutron-optical devices such as neutron guides, neutron polarizers, and neutron beam polarization analyzers. They are used in neutron studies of the structure of new materials, biological objects, quantum liquids, superconductors, etc. Neutron super-mirror polarizing elements are aperiodic multilayer structures made of alternating magnetic, based on Fe and Co, and non-magnetic, based on Ti and Zr, layers deposited by magnetron sputtering on glass or silicon substrates [1,2]. The polarizing and reflecting ability of such super-mirrors depends on the fine ratio of the thickness of the layers and the relative metal content in the layer.

The X-ray fluorescence (XRF) analysis has a number of significant advantages over other methods for determining the elemental composition of a substance. The first and probably the most significant advantage is the possibility of conducting an elemental analysis without taking into account the aggregate state and type of chemical compound of the sample. The second is the relative simplicity and unambiguity of the X-ray spectrum, which facilitate the interpretation of the results. The third feature of the X-ray spectral method is its non-destructive nature, i.e. the sample is not destroyed during the examination. The non-destructive nature of the analytical signal excitation makes it possible to analyze unique samples and samples, and, if necessary, repeat measurements under different conditions, but with the same sample.

A significant limitation of the use of XRF methods is the presence of strong matrix effects, i.e. the influence of the bulk of the sample on the signal extraction from small components of its complex composition. In this case, the matrix is a substrate material that differs significantly in its elemental composition (silicon) from the analyzed metal layers. Modern XRF techniques are based on well-developed methods and techniques for performing individual operations. A special place is occupied by the processing of measurement results. Here, the development of computing capabilities opens up new prospects for significantly reducing the detection limits of small impurities in samples.

The main objective of this study was to develop a methodology for determining the relative content of Fe, Co, and Ti, Zr in samples of super-mirrors using the X-ray fluorescence method with an errors of less than 1%, sufficient for confident control of their manufacturing technology.

2. X-ray fluorescence spectrometer with a radioisotope source

Our XRF-spectrometer uses a ring radioisotope source based on ¹⁰⁹Cd [3]. Its activity at the time of manufacture was 0.36 GBq, which ensures a photon flux into a solid angle of 10⁻⁴ sr of the order of 10³ s⁻¹. It is a source of X-ray radiation with an average energy of about 22 keV and gamma radiation with an energy of 88 keV (the latter cannot be considered as an intense source of gamma radiation, but requires special protection measures).

A fundamental feature of the design of the irradiation chamber in the spectrometer [4] is that the primary radiation from the source falls on the sample, but due to the collimator it does not enter the detector. The fluorescent radiation from the sample freely enters the detector. The variable height of the irradiation chamber ensures optimal conditions for irradiation and registration of secondary radiation, for which an HPGe-based detector is used. The detector's working area is 30 mm², and the energy resolution is 200 eV for the K α line of iron with an energy of 6.4 keV. The detector operates at a "nitrogen" temperature, so the volume of the irradiation chamber is separated by a beryllium window from the detector area. The latter is surrounded by a multilayer (aluminum-cadmium-lead) passive detector protection from the external radiation.

In order to determine the contribution of the source's primary radiation to the background, two series of measurements were carried out. In the first one, the spectrometer worked without a sample and measured the background directly. In the second series, the primary radiation from the source was partially directed into the detector. The recording time of the pure background was 2 hours and the radiation from the source was 1 hour. The corresponding X-ray spectra, reduced to one second, are shown in Fig. 1.

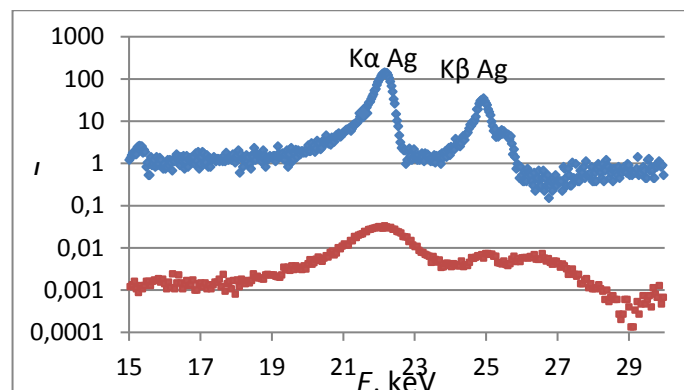


Fig. 1. Radiation spectra of the source (upper curve, blue dots) and background of the spectrometer (lower curve, red dots)

3. High-precision technique of XRF analysis

All XRF methods for determining the content of elements are based on comparing the line intensities of the characteristic X-ray spectra of the analyzed samples and reference samples. The so-called standard samples are often used as a reference sample. These are solids or liquids with precisely known elemental composition and concentrations, which are indicated in the sample passport. The error with which the content of the element in the reference sample is determined defines the systematic error of the method. It is generally assumed that this error should be no more than 1/3 of the error characterizing the reproducibility of the analysis method or its accuracy [5].

For the quantitative XRF analysis is most often used the direct method of the external standard [5,6]. The intensity of the analytical line is proportional to the concentration c_A^x of the detected element A in the sample:

$$I_x = \frac{K \cdot c_A^x}{(\mu_{m1}^x / \sin \varphi + \mu_{mi}^x / \sin \psi)} \quad (1)$$

For a reference sample –

$$I_0 = \frac{K \cdot c_A^0}{(\mu_{m1}^0 / \sin \varphi + \mu_{mi}^0 / \sin \psi)} \quad (2)$$

where

$$\mu_{m1} = c_A \mu_{m1}^A + (1 - c_A) \mu_{m1}^H \quad (3a)$$

$$\mu_{mi} = c_A \mu_{mi}^A + (1 - c_A) \mu_{mi}^H \quad (3b)$$

μ_{m1}^A, μ_{mi}^A – are the mass absorption coefficients of the atoms of the element being determined, respectively, of the primary radiation and the analytical line; μ_{m1}^H, μ_{mi}^H are the mass absorption coefficients of the packing atoms of the primary radiation and the analytical line. The angles φ and ψ are the angles of incidence of the primary radiation on the sample and the departure of the fluorescent radiation, respectively.

When determining the desired concentration using the direct method of the external standard, the intensity ratio is applied

$$\frac{I_x}{I_0} = \frac{c_A^x}{c_A^0} \cdot \frac{\mu_{m1}^0 / \sin \varphi + \mu_{mi}^0 / \sin \psi}{\mu_{m1}^x / \sin \varphi + \mu_{mi}^x / \sin \psi} \quad (4)$$

If the groups of the samples and the reference sample have the same chemical composition, and the concentration of the element to be determined in the samples varies little, then with a small assumption it can be assumed that the values of μ_{m1}^x and μ_{mi}^x are constant for all samples. With a reasonable choice of the concentration of element A in the comparison sample in relation to the concentrations of this element in the analyzed samples, the second multiplier on the right side of expression (4) is approximately equal to one, and the ratio is valid

$$c_A^x = \left(\frac{I_x}{I_0} \right) c_A^0 \quad (5)$$

Experimentally, the intensity is defined as the area of the analytical peak in the spectrum. The peak shape was approximated by the Gaussian function. The parameters of the analytical peaks, amplitude and width, were calculated using the method of the first and second moments. When processing the experimental spectra, the background was previously subtracted.

The errors are calculated using standard formulas. Statistical errors in analytical measurements are related to the discrete nature of secondary radiation detection, and the number of pulses in a given analyzer channel is a useful signal. The expected standard deviation (variance) of the total count is equal to the square root of the number of pulses N . When calculating the error of the intensity of the analytical line, which is equal to the area under the line in the spectrum, one speaks of the "full" standard deviation σ_t :

$$\sigma_t^2 = \sigma_N^2 + \sigma_1^2 + \sigma_2^2 + \sigma_3^2 + \dots \quad (6)$$

where σ_N is a statistical error, and σ_i are errors related to instrument error or calibration, operator actions, sample preparation, etc. These errors are often attributed to the background.

A fundamental feature of X-ray diffraction is the possibility of reducing the relative error and detection limits with increasing exposure time, i.e. increasing the statistics of X-ray spectra. Usually, the relative error for standard samples is much less than the error of the test sample. In this case, the latter is determined by the statistical error in determining the intensity of the analytical line.

4. Results

To determine the elemental composition of the neutron super-mirror, the state standard sample KS-1, salts of Ti and Sr, and metallic Zr in the form of powders were used as reference samples. The weight of the super-mirror sample was 19.2(1) mg. The time to set the spectra of the test sample was 10000 seconds. XRF spectra of the test and reference samples are shown in Fig. 2 and Fig. 3. The spectra are reduced to a registration time of 1 second. The results of the determination of the content of Mn, Fe, Co, Ti, Zr and Sr along the $K\alpha_1$ line in a sample of a neutron super-mirror are presented in Table 1. The relative content of the "working" metals Fe, Co, Ti,

and Zr (% by weight) in the neutron super-mirror, which showed the best properties, is shown in Table 2.

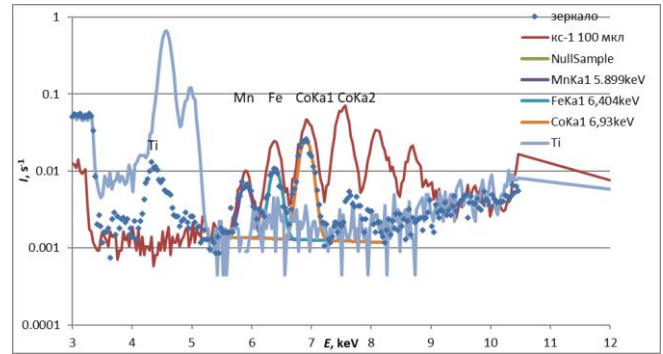


Fig. 2. Fluorescence X-ray spectra of a super-mirror sample and reference samples of Ti, Mn, Fe, Co ("soft" part).

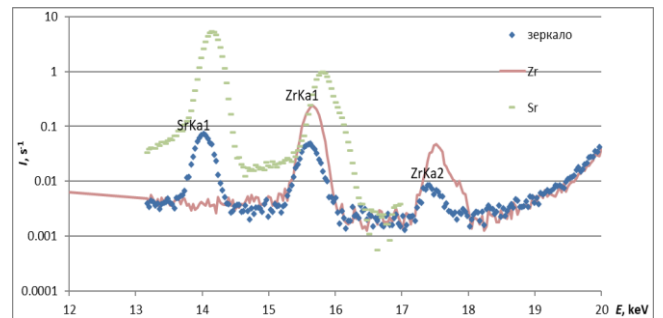


Fig. 3. Fluorescence X-ray spectra of a super-mirror sample and reference samples of Sr and Zr (the "hard" part)

Table 1. Metal content in the neutron super-mirror.

Element	Mn	Fe	Co	Ti	Zr	Sr
Concentration	0.16(5)	0.21(3)	0.54(3)	0.72(3)	0.114(3)	0.83(5)
C, % by weight	0.031(9)	0.040(6)	0.103(9)	0.14(2)	0.022(2)	0.16(1)

Table 2. The relative content of Fe, Co, Ti, and Zr in the super-mirror

Element	Fe	Co	Ti	Zr
Relative content, % by weight	13.25(3)	34.04(7)	45.5(1)	7.24(1)

5. Conclusions

The paper describes a precision technique for X-ray fluorescence determination of the content of Fe, Co, Ti, and Zr in neutron super-mirrors. The main part of this technique is the use of a spectrometer with a radioisotope source. The main factors influencing the result are the activity of the radioisotope source and the energy range of its radiation, the preparation of reference samples with the appropriate elemental composition, and the processing of secondary radiation spectra.

As a result, the following metal content was determined in a sample of a super-mirror with the best polarizing properties: Fe - 0.21(1)%; Co - 0.54(3)%; Ti - 0.72(3)%; Zr - 0.114(3) %. These results allowed us to determine the relative metal content in the super-mirror layers: Fe/Co - 28/72 and Ti/Zr - 86/14, with an error of less than 1%.

The principal conclusion of the work is that the proposed precision XRF technique makes it possible to control the process of magnetron sputtering of metals onto a substrate for the production of neutron polarizing super-mirrors with specified properties.

6. References

- [1] G.M. Drabkin et al. //JETPh. 1975. V.69. P.1916
- [2] N.K. Pleshanov. //Nucl. Instrum. Methods A. 2017.V.853. P.61.

- [3]** Catalog of radionuclide products of the scientific division of the Rosatom State Corporation. //SSC NIIAR, Dimitrovgrad, 2022 – 97 p.
- [4]** V.G. Zinoviev et al. The XRF spectrometer with a radioisotope source. //Preprint PNPI 3079, 2025 – 28 p.
- [5]** N.G. Chernorukov, O.V.Nipruk. Theory and practice of X-ray fluorescence analysis. //Nizhny Novgorod State University, Nizhny Novgorod. 2012 – 57 p.
- [6]** B. Beckhoff et al. Handbook of Practical X-Ray Fluorescence Analysis. "Springer", 2007–897 p.

Covered electrode selection for mma, optimal operating parameters and risk of cold cracking (hacc – hydrogen-assisted cold cracking)

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Abstract: This paper examines the critical criteria for selecting covered electrodes in manual metal arc (MMA) welding, focusing on achieving metallurgical compatibility with the base material and preventing welding defects. The performance characteristics, polarity requirements, and specific applications of major coating types—namely rutile, basic/low-hydrogen, and cellulosic electrodes—are evaluated alongside specialized consumables for stainless steels, cast iron, and hardfacing. Special attention is dedicated to the phenomenon of Hydrogen-Assisted Cold Cracking (HACC). Finally, the paper introduces the "Electrode advisor" digital application. By incorporating multi-criteria input parameters—including base material, geometry, thickness, welding position, power source polarity, and HACC risk factors—the software automates the selection of electrode classification, optimal diameter, and operating current. The integration of this application standardizes engineering decisions, streamlines quality assurance documentation, and significantly mitigates the risk of catastrophic weld failure in structural and marine applications.

Keywords: MMA WELDING, COVERED ELECTRODES, ELECTRODE SELECTION, HACC, HYDROGEN-ASSISTED COLD CRACKING, RUTILE, LOW-HYDROGEN, ELECTRODE ADVISOR APPLICATION.

1. Introduction

Covered electrodes are the main consumable for manual metal arc (MMA) welding. The coverings perform numerous technological and metallurgical functions, some of which are: easy striking and stable burning of the arc, creation of reliable slag and gas protection, modification and alloying of the weld metal, deoxidation, control of the cooling rate and many others [13]. The core wire of the electrode must correspond to the metal of the parts being welded and must provide the weld with mechanical and ductile properties that are not worse than those of the base metal.

"Correct selection" means the highest possible match between base material and weld metal (strength, hardness, elongation, impact toughness, corrosion resistance, etc.) and the absence of welding defects (cold cracks, undercuts, notches, bridging effect, ...). This depends on the correctly chosen: welding equipment (AC/DC), welding procedure (diameter and chemical composition of the electrode, welding current, arc length, travel speed, welding position, polarity), and control of diffusible hydrogen (properly selected electrode) [10], [6].

Covered electrodes for carbon (mild) steels are classified according to ISO 2560:2020 (system A/B by strength, impact toughness, type of coating and recovery) [1], [9], and for stainless steels – according to ISO 3581 (chemical composition of the deposited metal and type of coating) [2], [12].

The American AWS specifications use alphanumeric indices (e.g. E7018: E – electrode for manual metal arc welding; "70" – tensile strength 70 kpsi; "1" – all welding positions; "8" – basic/low-hydrogen coating and characteristic current/polarity) [10]. General terms and definitions are codified in AWS A3.0M/A3.0 [13].

2. Types of coatings and performance characteristics

Rutile (R; often E6013) – characterized by easy arc striking, smooth bead surface; suitable for thin sections in all welding positions, including vertical-down [8], [16].

Basic/low-hydrogen (B; E7018/E7016) – provide low diffusible hydrogen and high impact toughness; preferred for thick sections, shipbuilding/structural steels and low service temperatures [7], [12], [6].

Cellulosic (C; E6010/E6011) – deep penetration and high travel speed; typical for pipe root passes and site erection work [16], [10].

Special purpose – for stainless/heat-resisting steels to ISO 3581 [2], [12]; for hardfacing; for cast iron (Ni/NiFe) in cold / semi-hot welding.

3. Polarity and power source

Polarity affects the stability of the arc and the penetration capability of the electrode. Rutile electrodes can be classified as universal from the point of view of power source, working both with alternating and direct current (AC/DC, DC±) [8], [16].

Low-hydrogen (basic) E7018 electrodes are traditionally used on DC+ (DCEP). It should be noted that there are industrial formulations that also allow their use on alternating current (AC) [20], [3].

The choice of current is subject to the ranges given in the manufacturer's data sheet and is optimized for the welding position and diameter [16].

4. Control of diffusible hydrogen and the HxR designation

Cold cracks caused by diffusible hydrogen (HACC) occur after solidification and depend on the source of hydrogen (electrode, contaminated metal, etc.), hardness, microstructure and stresses. In carbon-manganese (C-Mn) steels the cracks often start in the heat-affected zone (HAZ) and may propagate into the weld metal [5]. Prevention includes the use of low-hydrogen electrodes, preheating, dry consumables and clean edges [6].

"H4/H4R" denotes a limited content of diffusible hydrogen (≤ 4 ml/100 g of deposited metal), and the letter "R" indicates additional resistance to moisture pick-up (27 °C / 80 % RH for 9 h) [11], [13].

5. Criteria for electrode selection (algorithm)

1) Material and mechanical requirements.

For S235–S355 (structural steels) and non-critical joints – rutile E6013; for thick sections, dynamic loading, low temperatures – low-hydrogen E7018 [1], [6]; for stainless steels – corresponding 308L/316L/309 class to ISO 3581 [2], [12].

2) Welding position.

All positions / thin material – rutile; vertical-up on thick sections – low-hydrogen; pipe roots – cellulosic [16], [10].

3) Power source and polarity.

Available AC source – rutile or AC-compatible E7018; for DC+ – low-hydrogen and rutile [16], [3].

4) Electrode diameter – selected according to thickness.

Practical guidelines (e.g. $\varnothing 2.5 \sim 2\text{--}5$ mm; $\varnothing 3.2 \sim 4\text{--}8$ mm; $\varnothing 4.0 \geq 8$ mm). For critical joints, the exact values are specified in the data sheet and WPS (Welding Procedure Specification) [16].

5) Hydrogen / cracking limitations.

At high carbon equivalent (CE) or high hardness – H4/H4R low-hydrogen electrodes, preheating and limited heat input in accordance with EN 1011-2 and good practice [6].

As examples from marine practice, the electrodes of manufacturers Unitor and Wilhelmsen can be cited –

LH-314N (LMA, low content of diffusible hydrogen, intended for shipbuilding / structural steels). The data sheet of these electrodes emphasizes their high impact toughness and crack resistance. They are intended for critical joints on "ship quality" steels [7].

GPO-302N (rutile, "general purpose"). Intended for use on low-carbon mild steels in all welding positions, including vertical-down; good bridging effect [8].

These examples illustrate the logic "E6013 for general repairs" versus "E7018 for critical joints / thick sections", in line with ISO/AWS classifications [1], [2], [10].

6. Defects in case of incomplete / wrong selection

- Cold cracks (HACC), often in the HAZ; their symptoms and orientation are described by TWI [5].
- Porosity, slag inclusions – incorrectly selected electrodes (moist electrodes, unsuitable coating for the welding position and/or low heat input – the amount of heat energy introduced per unit length of weld; it affects the HAZ and is usually specified in the WPS) – defined by the welding parameters [6].
- Lack of penetration – wrongly chosen electrode/coating for the specific needs (e.g. rutile instead of cellulosic for a root pass) or too low welding current values [16].

7. Storage, baking and exposure

After baking/drying, low-hydrogen electrodes should be stored in a holding oven at about 120 °C in order to prevent moisture pick-up [3].

Restoring the temperature regime when charging 20–80 kg of cold electrodes is slow (from 45 min to 2.5 h) and has to be taken into account when organizing the work [9].

"R"-classified (H4R) products are designed for increased moisture resistance, but the manufacturer's recommendations and exposure limits still have to be respected [11], [12].

Indicative drying regimes for electrodes:

- E7018 / E7016 (low-hydrogen, H4/H4R) – (260–350) °C for 1–2 h.
- E6013 (rutile) – (100–150) °C for 0.5–1 h (if there is moisture) – high temperatures are not used so as not to damage the coating.
- E6010 / E6011 (cellulosic) – not baked.
- Stainless/nickel electrodes 308L, 316L, Ni/NiFe – (200–300)°C for 1÷2 h.

8. Parameters: current, speed, welding technique

Industrial guidelines (e.g. Lincoln) provide operating ranges by type and diameter, as well as recommendations on polarity and control of arc blow, especially for DC [16].

In practice, the welding current for Ø2.5 / 3.2 / 4.0 mm electrodes usually falls around ~60–90 A / ~90–130 A / ~120–170 A and is fine-tuned according to position, workpiece and coating type, always validated against data sheet / WPS [16].

Figures 1, 2 and 3 show recommended welding current values depending on the thickness of the part being welded, respectively for E6013 (rutile), E7018 (basic) and E6010 (cellulosic) electrodes.

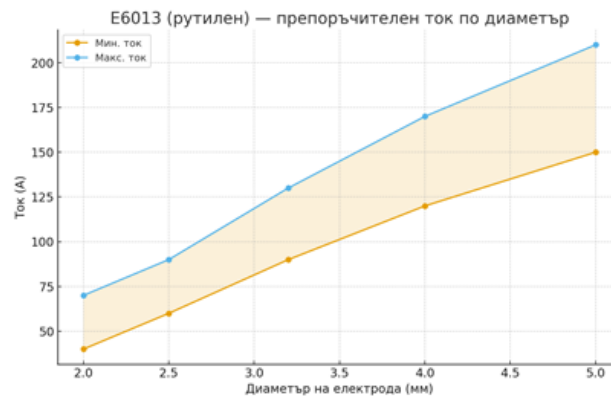


Fig. 1 E6013 (rutile): recommended current by diameter [14], [16]

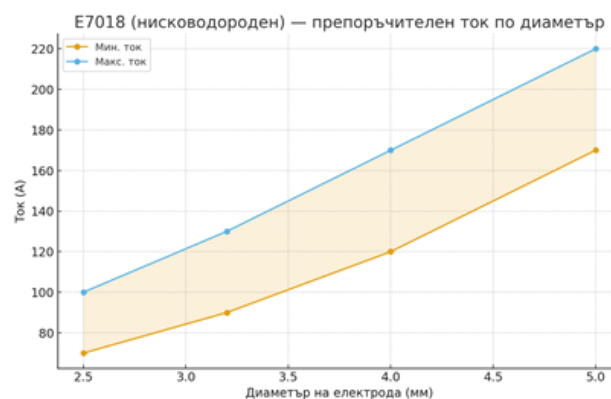


Fig. 2 E7018 (low-hydrogen): recommended current by diameter [14], [16].

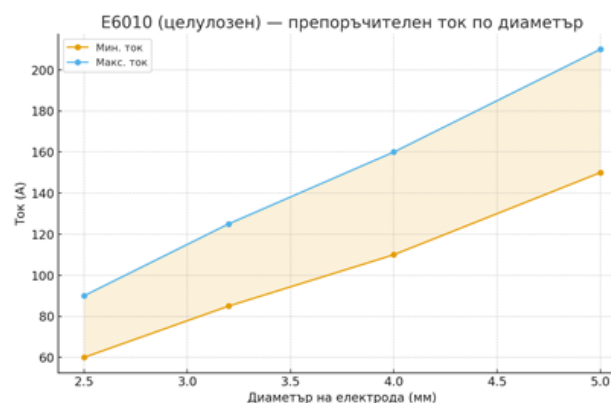


Fig.3 E6010 (cellulosic): recommended current by diameter [14], [16].

9. Cold cracks HACC (hydrogen-assisted cold cracking)

Cold cracks caused by the presence of diffusible hydrogen appear after completion of the welding process at approximately room temperature; most often this occurs in the HAZ or in the weld metal.

The three necessary conditions for this defect are:

- Diffusible hydrogen in the weld/HAZ (caused by moisture, residues of oils, paint, use of rutile or cellulosic electrodes, insufficient baking of electrodes).

- Brittle and hard microstructure (high carbon equivalent CE, large material thickness with martensitic structure and high hardness).
- Presence of tensile stresses (restraint caused by inappropriate welding sequence, rapid cooling).

To control these three components (hydrogen, hard structure, stresses), we propose the following checklist in tabular form for managing the risk of undesired cold cracks.

Mark "Yes" where a risk is present; ≥ 3 "Yes" answers $\Rightarrow$ take measures (E7018 H4/H4R; preheating; dry electrodes; control of interpass temperature; lower HAZ hardness) [5], [6], [11], [12].

Cold cracks (HACC – Hydrogen-Assisted Cold Cracking) – checklist:

Table 1: Risk factors.

Tick	Risk factor	Notes
☐	High carbon equivalent (CE) of the steel or thickness above 15 mm.	
☐	High restraint / fixation of the part (restricted shrinkage).	
☐	Low ambient temperature / no preheating.	
☐	Contaminated edges (moisture, oils, paint, rust).	
☐	Moist electrodes.	

Table 2: Measures when risk is identified

Tick	Measure	Notes
☐	Low-hydrogen electrodes (e.g. E7018 H4/H4R) + correct baking/storage.	
☐	Preheating according to WPS and control of interpass temperature.	
☐	Cleaning of edges to bare, dry metal (no paint/rust/oils).	
☐	Reduction of restraint and optimization of weld sequence.	
☐	Control of heat input and travel speed within the WPS limits.	

10. Electrode selection.

Table 3: Matrix – guideline for electrode selection (carbon steel, pipe root)

Material	Carbon steel S235–S355	Carbon steel (thick ≥ 8 mm)	Pipe root (C-Mn)
Position/ thickness	Flat/fillet, thin ≤ 5 mm	Vertical-up/positional	Vertical-up/-down
Power source	AC or DC $\pm$	DC+	DC+
Recommendation	E6013 (rutile) $\varnothing 2.0$ – 2.5 ; 60–90 A	E7018 (low-hydrogen) $\varnothing 3.2$ – 4.0 ; 110–160A	E6010 $\varnothing 2.5$ – 3.2 ; 85–125 A
basis/ source	Easy arc striking, smooth bead	Strength/ impact toughness; low H	Deep penetration

Table 4: Matrix – guideline for electrode selection (stainless steel, dissimilar)

Material	Stainless 304 $\leftrightarrow$ 304	Stainless 316 $\leftrightarrow$ 316	Dissimilar 304/316 $\leftrightarrow$ carbon steel
Position/	All positions	All positions	All positions

thickness			
Power source	AC/DC	AC/DC	AC/DC
Recommendation	308L (stainless)	316L (stainless)	309/309 Mo
basis/ source	Matching composition	Mo for corrosion resistance	Buffer composition for dissimilar joints

Table 5: Matrix – guideline for electrode selection (cast iron, low temperature high CE)

Material	Cast iron (repair)	Low temperature / high CE
Position/ thickness	Short runs	Critical joints
Power source	AC/DC	DC+
Recommendation	Ni or NiFe	E7018 H4/H4R
basis/ source	Minimization of cracking; machinability	Control of H; moisture resistance

11. Application for selecting a suitable electrode

On the basis of everything discussed so far, with the aim of fast and correct selection of a suitable electrode, a quick-selection application – "Electrode advisor" – was developed.

Electrode Selection Advisor (SMAW)
Results are indicative. Validate against WPS and manufacturer data sheets (E6013/E7018/E6010; 308L/316L/309Mo; Ni/NiFe).

☒ Expert Mode (positional current correction and recommended setpoint)

1. Material

Dissimilar: Stainless $\leftrightarrow$ Carbon Steel

2. Parameters & Power

Thickness (mm): 6 Position: Horizontal

Power Source: AC Criticality: General Repairs

3. HACC Risk (Hydrogen-Assisted Cold Cracking)

☐ High CE/Hardness or Thickness ≥ 15 mm ☐ High Restraint / High Stresses ☐ Low Ambient Temperature, No Preheat

☐ Dirty Edges (Moisture/Oil/Paint) ☐ Non-Low-Hydrogen Process/Consumable

Total factors: 0 Risk: LOW

Recommended Selection

CLASS (AWS): **309/309Mo** DIAMETER: **3.2 mm** CURRENT (GUIDELINE): **86–124 A**

Polarity: AC

EXPERT MODE – CURRENT CALCULATION

Base Range (Flat): 90–130 A Position Correction (Horizontal): -5% Recommended Range: 86–124 A

Optimal Setpoint: **105 A**

RATIONALE

- High Cr/Ni acts as a buffer for dissimilar joints.

Fig.4 Screenshot of the quick-selection application "Electrode advisor"

The application uses the following input data, in the form of drop-down menus:

- Base material of the part:
 - Carbon steel (S235–S355);
 - Shipbuilding/structural steel (more critical joints);
 - Stainless 304 / 316;
 - Dissimilar joint (stainless steel – carbon steel);
 - Cast iron (with option "machinability required" – Ni vs NiFe);

- Hardfacing (general guideline).
- Geometry and position
 - Thickness (mm);
 - Welding position: flat / horizontal / vertical-up / vertical-down / overhead;
- Power source / polarity
 - AC, DC+ (DCEP) or DC- (DCEN).
- Criticality of the task
 - General repair vs critical/structural.
- HACC-factors (assessment of hydrogen-assisted cold cracking risk)
 - High carbon equivalent / hardness or thickness ≥ 15 mm;
 - High restraint / restricted deformations;
 - Low ambient temperature without preheating;
 - Contaminated edges (moisture/oils/paint);
 - Use of non-low-hydrogen process/consumable.
- (Cast iron only) Is machinability of the weld required?
 - Determines the choice Ni (better machinability) versus NiFe (higher strength).

The application automatically performs the following operations:

- Calculates HACC risk (low / medium / high) and gives recommendations;
- Selects electrode class (E6013/E7018/E6010; 308L/316L/309Mo; Ni/NiFe);
- Proposes electrode diameter according to part thickness and welding position;
- Indicates approximate current according to diameter and electrode class;
- Adds recommendations on polarity (e.g. "DC+ (DCEP)" for E7018/E6010; "AC/DC±" for E6013).

Conclusions:

The application converts a complex engineering and technological choice into a structured and explained process by:

1. Showing the correct electrode/diameter/polarity for the context.
2. Managing HACC risk with the help of a checklist and giving guidance on its minimization.
3. Generating documents for QA/WPS archiving.
4. Standardizing practice and speeding up the work of the team.

3. References

1. A. Mecke, I. Lee, J.R. Baker jr., M.M. Banaszak Holl, B.G. Orr, Eur. Phys. J. E 14, 7 (2004)
2. ISO 3581:2023 — Covered electrodes for MMA welding of stainless and heat-resisting steels
3. TWI — Welding Consumables — Part 2 (storage in holding oven ~ 120 °C)
4. TWI FAQ — Baking time/thermal inertia for 20–80 kg batches
5. TWI — Hydrogen Cracks in Steels: Identification
6. TWI — Hydrogen Cracks in Steels: Prevention and Best Practice
7. Wilhelmsen/UNITOR — LH-314N (LMA) product sheet
8. Wilhelmsen/UNITOR — GPO-302N product sheet
9. ELGA Guide — EN ISO 2560-A symbols and current/recovery indications
10. Hobart Brothers — Electrode Classification (E7018, etc.)
11. ESAB — H4R explanation (≤ 4 ml/100 g; moisture resistance criteria)
12. Washington Alloy — E7018 H4R datasheet
13. AWS A3.0M/A3.0:2020 — Terms and Definitions
14. Lincoln Electric — Stick Electrode Welding Guide
15. Lincoln Electric — 7018 AC product page
16. UNITOR range overview — maintenance electrodes

Transformation of waste polystyrene into high surface area activated carbon: structural and surface insights

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Abstract: Activated carbon was prepared from waste polystyrene through acid treatment and pyrolysis to obtain a porous adsorbent with controlled structural features. Nitrogen adsorption reveals a type IV isotherm with H4 hysteresis and a high specific surface area of 1382 m²/g, indicating combined micro- and mesoporosity. SEM imaging shows irregular carbon agglomerates with a highly textured surface, while elemental and FTIR analyses confirm an aromatic carbon matrix with limited heteroatom functionality. These structural and chemical features demonstrate that the synthesized carbon material is well-developed and suitable for further application-oriented studies, including detailed adsorption performance evaluation.

Keywords: ACTIVATED CARBON, POLYSTYRENE WASTE, PYROLYSIS, SURFACE CHARACTERISTICS

1. Introduction

Plastic waste has become one of the most persistent environmental challenges of modern society [1]. The continuous growth in global plastic production, combined with limited recycling capacity, results in the accumulation of large quantities of polymeric materials in landfills and natural ecosystems. Polystyrene, widely used in packaging and consumer products, is particularly problematic due to its high consumption rate, low biodegradability, and the scarcity of efficient recycling routes. Once released into the environment, polystyrene fragments slowly degrade into microplastics, which disperse through soil and water systems and may enter biological food chains. Their presence has raised concerns about potential ecological toxicity and long-term impacts on human health [2].

Beyond environmental persistence, the production and disposal of plastics contribute significantly to greenhouse gas emissions, reinforcing the need for sustainable waste-management strategies. In recent years, the concept of converting polymeric waste into value-added carbon materials has gained attention as a promising approach to both waste reduction and resource recovery. Polystyrene, with its aromatic backbone and high carbon content, is a suitable precursor for producing activated carbon through controlled thermochemical treatment [3, 4].

Transforming waste polystyrene into activated carbon not only mitigates environmental pollution but also provides access to porous materials with desirable structural and surface characteristics. Such carbons are relevant for adsorption, catalysis, energy storage, and other technological applications [4].

The present study focuses on the synthesis and characterization of activated carbon obtained from waste polystyrene through acid pretreatment and pyrolysis. The aim is to examine the development of porosity, morphology, and surface chemistry in the resulting material and to establish its potential for further application-oriented research.

2. Materials and Methods

2.1. Preparation of the Carbon Material

The activated carbon was synthesized through a two-stage thermochemical conversion process involving acid-induced oxidation followed by high-temperature pyrolysis. In the first stage, the waste polystyrene was melted and subjected to oxidation with concentrated sulfuric acid at a 1:1 weight ratio. The mixture was maintained at 350 °C under continuous stirring until a solid carbonaceous product was formed. This treatment promoted extensive structural rearrangement and partial aromatization of the polymer matrix, preparing the material for subsequent carbonization.

In the second stage, the obtained solid was pyrolyzed at 900 °C in a carbon dioxide atmosphere. The temperature was increased at a controlled heating rate of 10 °C/min, and the sample was held at the

final temperature to ensure complete thermal decomposition and pore development. After cooling to room temperature under CO₂ flow, the resulting carbon material was collected, and stored for further characterization. This combined oxidation-pyrolysis route enabled the formation of a porous, predominantly aromatic carbon structure suitable for detailed textural and surface analysis.

2.2. BET Surface Area and Porosity Characterization

Nitrogen adsorption-desorption isotherms at 77 K were recorded to evaluate the specific surface area and textural properties of the carbon material. Prior to analysis, the samples were degassed under vacuum at elevated temperature to remove adsorbed moisture and volatile species. The amount of nitrogen adsorbed on the carbon surface was measured over a range of relative pressures, and the specific surface area was calculated according to the Brunauer-Emmett-Teller (BET) method. Pore size distribution and total pore volume were obtained from the desorption branch using the Barrett-Joyner-Halenda (BJH) model. Measurements were performed using a Quantachrome gas sorption analyzer.

2.3. SEM/EDS analysis

Scanning electron microscopy (SEM) was used to examine the morphology of the carbon material, including particle structure, surface texture and pore development. The samples were mounted on conductive stubs and sputter-coated to minimize charging during imaging. Elemental composition was determined by energy-dispersive X-ray spectroscopy (EDS) integrated into the SEM system, providing qualitative and quantitative information on the main elements present in the material.

2.4. FTIR analysis

Fourier-transform infrared (FTIR) spectroscopy was used to identify the surface functional groups present in the carbon material. Spectra were recorded in the mid-infrared region, and characteristic absorption bands were analyzed to evaluate the presence of aromatic structures, oxygen-containing functionalities and other groups formed or modified during the oxidation and pyrolysis steps.

2.5. Raman analysis

Raman spectroscopy was employed to assess the structural ordering of the carbon material. The spectra were used to evaluate the relative intensity of the D and G bands, providing insight into the degree of graphitization, defect density and the overall structural evolution resulting from the thermochemical conversion of polystyrene.

3. Results and Discussion

3.1. Textural Properties

N₂ adsorption at 77 K reveals a type IV isotherm with an H4 hysteresis loop, characteristic of materials combining microporosity with slit-like mesopores (Fig.1) [5]. The steep increase in adsorbed volume at low relative pressures ($p/p_0 < 0.05$) indicates well-

developed microporosity, while the presence of hysteresis confirms the contribution of mesopores to the overall pore structure.

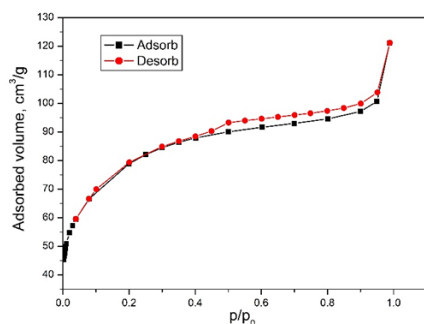


Fig. 1 N_2 adsorption-desorption isotherm at 77.4 K for the activated carbon

The carbon material exhibits a highly developed and hierarchically organized pore structure (Table 1). The exceptionally high specific surface area (1382 m²/g) confirms the dominant contribution of micropores, which account for approximately 41% of the total pore volume. Mesopores and macropores form comparable fractions of the structure, supporting efficient diffusion and enhanced mass transport. Overall, the combination of micro-, meso- and macropores provides both strong adsorption potential and favorable transport properties, characteristic of carbon materials produced through acid oxidation followed by high-temperature CO₂ activation.

Table 1: Specific surface area and textural parameters of the activated carbon

Textural parameter	Measured value
Total pore volume, cm ³ /g	1.025
Micropore volume, cm ³ /g	0.424
Mesopore volume, cm ³ /g	0.284
Macropore volume, cm ³ /g	0.317
BET specific surface area, m ² /g	1382

3.2. Morphology and Elemental Composition

The morphology of the activated carbon was examined by scanning electron microscopy (Figure 2). The SEM images reveal irregular, fragmented particles with a rough and highly textured surface. Numerous surface irregularities, fissures and pore openings are visible, consistent with the hierarchical pore structure identified through nitrogen adsorption. This morphology is typical for activated carbons obtained through thermochemical conversion of polymeric precursors.

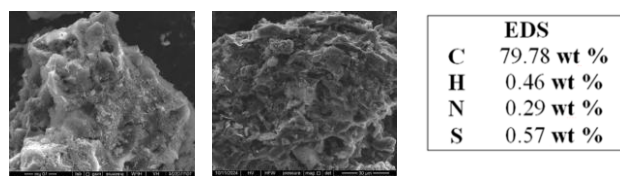


Fig. 2 SEM morphology and EDS elemental composition of the activated carbon

The elemental composition (Fig. 2) is dominated by carbon (79.78%), confirming efficient carbonization of the polystyrene precursor. The very low hydrogen (0.46%) and nitrogen (0.29%) contents are characteristic of highly aromatized carbon structures formed during high-temperature pyrolysis. A minor sulfur content (0.57%) reflects the acid treatment step and suggests the presence of sulfur-containing surface functionalities. Overall, the elemental profile aligns with the observed morphology and the textural parameters derived from nitrogen adsorption, supporting the

conclusion that the material is extensively carbonized and possesses a well-developed porous structure.

3.3. FTIR spectrum of the activated carbon

The FTIR spectrum of the activated carbon (Fig. 3) displays weak and broad absorption bands, typical of highly carbonized materials with a limited number of surface functional groups. The broad band at 3435 cm⁻¹ corresponds to O–H stretching vibrations, most likely associated with adsorbed moisture. The weak peaks in the 2969 – 2852 cm⁻¹ region arise from residual aliphatic C–H groups. The band at 1631 cm⁻¹ is attributed to aromatic C=C vibrations and contributions from adsorbed water.

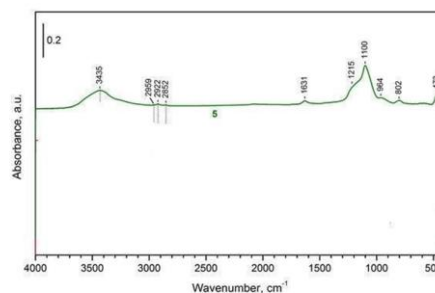


Fig. 3 FTIR spectrum of the activated carbon

Several bands in the 1215 – 1100 cm⁻¹ region indicate C–O stretching in oxygen-containing functionalities and may also reflect S–O/S=O vibrations introduced during H₂SO₄ treatment, consistent with the elemental analysis. The peaks at 964 and 802 cm⁻¹ correspond to out-of-plane deformations of aromatic C–H bonds, while the low-frequency band at 473 cm⁻¹ is associated with skeletal vibrations of the carbon matrix [6].

Overall, the FTIR spectrum confirms the presence of an aromatized carbon structure containing small amounts of oxygen- and sulfur-bearing groups, in agreement with the high degree of carbonization indicated by the textural, morphological and elemental analyses.

3.4. Raman analysis of the activated carbon

The Raman spectrum of the activated carbon (Fig. 4) is characterized by two prominent bands: the D band at ~1350 cm⁻¹ and the G band at ~1580 – 1600 cm⁻¹, accompanied by a weaker and broader 2D band near 2700 cm⁻¹. The relatively high intensity ratio I_D/I_G indicates a substantial degree of structural disorder and the presence of small graphitic domains. This spectral profile is typical of turbostratic carbon, where graphene layers are misaligned and only weakly ordered.

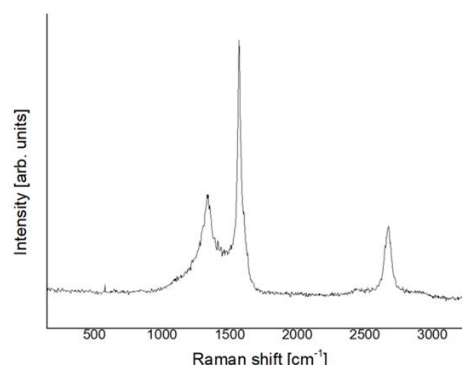


Fig. 4 Raman spectrum of the activated carbon

The Raman results are consistent with the textural data, which reveal a high specific surface area and a well-developed porous structure. Overall, the spectrum confirms that the material is a defect-rich, turbostratic carbon with low crystallinity and an extended network of sp²-hybridized domains, supporting its potential for adsorption and catalytic applications [7, 8].

4. Conclusion

The comprehensive characterization of the activated carbon demonstrates that the applied synthesis route – acid treatment of the polystyrene precursor followed by high-temperature CO₂ activation – successfully produces a highly porous, defect-rich carbon material with well-developed surface functionality. Nitrogen adsorption measurements reveal a hierarchical pore structure dominated by micropores and complemented by meso- and macropores, resulting in a high specific surface area and favorable mass-transport properties. SEM imaging confirms the presence of irregular, fragmented particles with a rough and textured surface, while EDS analysis verifies efficient carbonization and the incorporation of oxygen- and sulfur-containing groups originating from the oxidation step.

FTIR spectroscopy further supports the formation of an aromatized carbon matrix enriched with limited but identifiable oxygen- and sulfur-bearing functionalities. Raman analysis indicates a turbostratic, defect-rich structure with a high I_D/I_G ratio, consistent with small graphitic domains and low crystallinity. Together, these findings confirm that the obtained material is a highly disordered, microporous activated carbon with an extended network of sp²-hybridized domains.

Overall, the structural, morphological and spectroscopic results demonstrate that the synthesized activated carbon possesses properties favorable for adsorption and catalytic applications, including high surface area, hierarchical porosity, abundant defect sites and tailored surface chemistry. These characteristics highlight its potential for use in processes requiring efficient mass transfer, strong adsorptive interactions and tunable surface reactivity.

4. References

1. R. Geyer, J.R. Jambeck, K.L. Law, *Sci. Adv.* **3**, e1700782 (2017)
2. S.L. Wright, F.J. Kelly, *Environ. Sci. Technol.* **51**, 6634–6647 (2017)
3. L. Pereira, V. Castillo, M. Calero, G. Blázquez, R.R. Solís, M.A. Martín-Lara, *J. Water Process Eng.* **62**, 105386 (2024)
4. Q. Hou, Y. Zhang, C. Wang, *J. Environ. Chem. Eng.* **13**, 115368 (2025)
5. M. Thommes, K. Kaneko, A.V. Neimark, J.P. Olivier, F. Rodriguez-Reinoso, J. Rouquerol, K.S.W. Sing, *Pure Appl. Chem.* **87**, 1051–1069 (2015)
6. M.-M. Titirici, R.J. White, C. Falco, M. Sevilla, *Energy Environ. Sci.* **5**, 6796–6822 (2012)
7. A.C. Ferrari, J. Robertson, *Philos. Trans. R. Soc. Lond. A* **362**, 2477–2512 (2004)
8. D.B. Schuepfer, F. Badaczewski, J.M. Guerra-Castro, D.M. Hofmann, C. Heiliger, B. Smarsly, P.J. Klar, *Carbon* **161**, 359–372 (2020)

Multi-parameter assessment of the relative electrochemical condition of reinforced concrete structures exposed to stray currents and chloride environments

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Abstract. *The long-term reliability of reinforced concrete structures is strongly influenced by electrochemical deterioration processes affecting embedded steel reinforcement. In addition to conventional environmental actions, stray electrical currents may disturb the passive state of reinforcing steel and contribute to the initiation or acceleration of corrosion, particularly when combined with chloride-contaminated environments. This study proposes a multi-parameter framework for assessing the electrochemical reliability of reinforced concrete structures subjected to combined electrical and environmental actions. The assessment is based on three principal electrochemical indicators: reinforcement corrosion potential, electrical resistivity of concrete and corrosion current density. Five representative exposure scenarios are considered, ranging from a reference condition without significant electrical influence to combined exposure involving increased moisture, chlorides and simulated stray currents. The selected parameters are integrated into a dimensionless Electrochemical Reliability Index (ERI), developed to enable comparative classification of structural condition and deterioration risk. The proposed methodology combines normalised electrochemical indicators with a weighted deterioration model and sensitivity analysis. For the baseline weighting system, the calculated ERI values decrease from 1.000 for the reference K0 condition to 0.846 for E1, 0.651 for E2, 0.374 for E3 and 0.000 for the highly aggressive E4 scenario. The most pronounced reliability reduction occurs between moderate electrical influence and simulated stray-current exposure. Sensitivity analysis confirms that the ranking $K0 > E1 > E2 > E3 > E4$ remains unchanged under three weighting systems. The proposed framework is intended as a comparative engineering assessment tool rather than a substitute for detailed structural inspection or standardised corrosion diagnosis. Its principal contribution lies in integrating complementary electrochemical parameters into a unified reliability-oriented indicator that may support condition assessment, maintenance prioritisation and durability management of reinforced concrete structures exposed to complex electrical and chloride environments.*

KEYWORDS: REINFORCED CONCRETE; STRAY CURRENTS; REINFORCEMENT CORROSION; CHLORIDE EXPOSURE; ELECTRICAL RESISTIVITY; CORROSION CURRENT DENSITY; ELECTROCHEMICAL RELIABILITY; DURABILITY.

1 Page layout

Reinforced concrete structures are generally designed for long service lives; however, their durability and reliability may be significantly reduced by reinforcement corrosion. Corrosion leads to progressive loss of steel cross-section, deterioration of the steel-concrete bond, cracking and spalling of the concrete cover and, at advanced stages, reduction in load-bearing capacity and structural safety. Recent experimental investigations confirm that corrosion-related deterioration becomes particularly critical when reinforced concrete is exposed to combinations of cracking, aggressive ions and external electrical influences [1].

Among the environmental actions responsible for reinforcement depassivation, chloride ingress represents one of the most important durability threats. Once a sufficient concentration of chlorides reaches reinforcement level, the passive condition of embedded steel may be destabilised and localised corrosion may develop. The deterioration process becomes more complex in structures located near electrified railway systems, metro infrastructure and other direct-current installations, where leakage of electrical current from the intended return circuit may generate stray-current fields in surrounding soil and conductive construction materials [2]. Experimental studies have shown that stray-current corrosion differs from conventional constant direct-current acceleration and that the electrical characteristics of the current may influence both electrochemical reactions and chloride electromigration [2].

The interaction between stray electrical currents and chloride-contaminated environments is particularly important because the two actions may not operate independently. Investigations of concrete exposed to stray current have demonstrated that electrical effects can modify the transport behaviour of chloride ions and affect the interfacial transition zone between aggregate and cement matrix. Chen et al. observed changes in ITZ thickness and porosity under stray-current conditions and reported enhanced chloride transport through this microstructurally sensitive region [3]. These findings indicate that durability assessment of reinforced concrete structures located in electrically influenced environments should account not only for conventional diffusion processes but also for electrically induced ion migration and microstructural deterioration.

A further complication arises when cracks are present in the concrete cover. Cracks provide preferential transport paths for moisture and aggressive ions and simultaneously modify local electrical properties. Experimental and numerical research has demonstrated a pronounced coupling between cracking and stray-current exposure, leading to asymmetric distributions of electrical fields and ionic concentrations around reinforcement [1]. This behaviour illustrates the need for diagnostic methods capable of identifying deterioration before substantial structural damage becomes visible.

Electrochemical measurements provide an important basis for condition assessment. Reinforcement corrosion potential can be measured in accordance with ASTM C876-22b, which covers estimation of the electrical corrosion potential of uncoated reinforcing steel in field and laboratory concrete. However, potential measurements alone do not provide a complete structural diagnosis; complementary information such as corrosion rate, chloride content and environmental exposure conditions is often necessary [4]. This limitation is particularly relevant in the presence of stray currents, where externally induced electrical fields may affect individual electrochemical indicators.

Corrosion current density determined through polarisation-resistance measurements provides complementary information on instantaneous corrosion activity. RILEM TC 154-EMC recommendations relate polarisation resistance to corrosion current density and encourage interpretation together with corrosion potential, concrete resistivity and other exposure parameters [5]. Electrical resistivity of concrete is also important because it reflects the ability of the cementitious matrix to support ionic current flow. Low resistivity generally facilitates electrochemical current transmission, whereas higher resistivity may restrict corrosion kinetics. Nevertheless, resistivity is strongly influenced by moisture, temperature, pore structure and ionic concentration and should therefore be interpreted as part of a multi-parameter diagnostic framework.

The importance of controlled electrochemical monitoring is also reflected in international standards related to corrosion protection. ISO 12696:2022 specifies performance requirements for cathodic protection of steel embedded in cement-based concrete in new and existing structures and requires appropriate performance

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monitoring of protected systems [6]. Although the standard is focused on cathodic protection rather than stray-current diagnosis, it demonstrates the broader engineering importance of electrochemical monitoring when steel in concrete is exposed to electrically influenced environments.

Despite significant advances in electrochemical testing, assessment of reinforced concrete structures exposed to simultaneous stray-current and chloride effects remains challenging. Individual diagnostic parameters may provide different indications of structural condition because each represents a different component of the corrosion process. Corrosion potential primarily reflects the electrochemical state, resistivity characterises electrical and ionic conductivity of surrounding concrete, while corrosion current density provides information related to corrosion kinetics. Therefore, an integrated interpretation of these indicators is required for comparative reliability assessment.

Accordingly, the objective of the present study is to develop a multi-parameter methodology for evaluating the electrochemical reliability of reinforced concrete structures exposed to different levels of electrical and environmental influence. Five representative exposure scenarios are considered, ranging from a reference condition without significant electrical disturbance to a highly aggressive combined condition involving increased moisture, chlorides and simulated stray currents. The proposed methodology integrates reinforcement potential, concrete electrical resistivity and corrosion current density into a dimensionless Electrochemical Reliability Index (ERI). The ERI is introduced as an engineering research index for comparative analysis and sensitivity assessment; it is not proposed as a replacement for standardised corrosion diagnostic procedures.

2 Methodology

2.1 Research Framework and Definition of Exposure Scenarios

The proposed methodology was developed to evaluate the comparative electrochemical reliability of reinforced concrete structures subjected to different combinations of electrical and environmental actions. Five representative exposure scenarios were defined, ranging from a reference condition without significant electrical disturbance to a combined high-aggressiveness scenario involving increased moisture, chloride contamination and simulated stray-current influence.

Table 1. Definition of the investigated exposure scenarios

Group	Exposure conditions	Principal purpose
K0	Reference condition without significant external electrical influence	Definition of baseline electrochemical behaviour
E1	Low-level electrical influence	Assessment of minor electrical disturbance
E2	Moderate electrical influence	Simulation of prolonged operating conditions
E3	Simulated stray-current exposure	Assessment of electrically accelerated electrochemical deterioration
E4	Increased moisture + chlorides + simulated stray currents	Assessment of the most aggressive combined exposure scenario

The K0 group represents the reference state of reinforced concrete under conditions in which reinforcement remains predominantly protected by the alkaline concrete environment and no significant

externally imposed electrical disturbance is considered. The E1 and E2 groups represent progressively increasing electrical influence. The E3 group represents simulated stray-current exposure, while E4 combines stray currents with increased moisture and chloride presence. The adopted sequence of expected exposure severity is $K0 < E1 < E2 < E3 < E4$.

2.2 Selected Electrochemical Parameters

Three complementary parameters were selected for the reliability assessment: reinforcement corrosion potential, E_{corr} [mV]; electrical resistivity of concrete, ρ [k Ω ·cm]; and corrosion current density, i_{corr} [μ A/cm²]. Corrosion potential provides information related to electrochemical state, resistivity characterises the capacity of the surrounding cementitious matrix to conduct ionic current, and corrosion current density represents the kinetic component of electrochemical deterioration. The central hypothesis is that a combined interpretation provides a more reliable comparative description than any single indicator considered independently.

2.3. Normalisation of the Electrochemical Parameters

Because the selected indicators are expressed in different physical units and their unfavourable directions differ, each parameter is transformed into a dimensionless deterioration indicator between 0 and 1. A value of 0 represents the most favourable condition within the analysed dataset, while 1 represents the least favourable condition.

$$R_E = (E_{\max} - E_i) / (E_{\max} - E_{\min}) \quad (1)$$

$$R_p = (\rho_{\max} - \rho_i) / (\rho_{\max} - \rho_{\min}) \quad (2)$$

$$R_i = (i_{\text{corr},i} - i_{\text{corr},\min}) / (i_{\text{corr},\max} - i_{\text{corr},\min}) \quad (3)$$

This study-specific normalisation is intended for comparative analysis of the investigated scenarios and does not replace standardised corrosion-potential, resistivity or corrosion-rate criteria.

2.4 Electrochemical Deterioration Index and Electrochemical Reliability Index

The three normalised parameters are integrated into an Electrochemical Deterioration Index (EDI). The general formulation is a weighted linear combination, where the weighting factors sum to unity.

$$EDI = w_E R_E + w_p R_p + w_i R_i \quad (4)$$

$$w_E + w_p + w_i = 1 \quad (5)$$

For the baseline model, the weighting coefficients are set to $w_E = 0.30$, $w_p = 0.25$ and $w_i = 0.45$. A somewhat greater weight is assigned to corrosion current density because it is used as the principal kinetic indicator of ongoing corrosion activity, whereas potential and resistivity provide complementary information. These weighting factors are author-proposed modelling assumptions and are subsequently examined through sensitivity analysis.

$$EDI = 0.30 R_E + 0.25 R_p + 0.45 R_i \quad (6)$$

$$ERI = 1 - EDI \quad (7)$$

$$ERI = 1 - (0.30 R_E + 0.25 R_p + 0.45 R_i) \quad (8)$$

The Electrochemical Reliability Index therefore varies theoretically from 0 to 1. Values approaching 1 indicate comparatively favourable electrochemical reliability, whereas values approaching 0 indicate severe electrochemical deterioration within the investigated comparative framework.

2.5 Proposed Reliability Classification

Table 2. Study-specific comparative classification of ERI values

ERI range	Reliability class	Interpretation
$0.80 \leq \text{ERI} \leq 1.00$	Very high	Favourable electrochemical condition
$0.60 \leq \text{ERI} < 0.80$	High	Generally favourable condition with limited deterioration indicators
$0.40 \leq \text{ERI} < 0.60$	Moderate	Transitional condition requiring increased attention
$0.20 \leq \text{ERI} < 0.40$	Low	Significant electrochemical deterioration risk
$0.00 \leq \text{ERI} < 0.20$	Very low	Severe or critical comparative electrochemical condition

These limits are introduced exclusively for internal comparative interpretation of the proposed model. They do not represent standardised acceptance limits and should not be used independently to declare an existing structure safe or unsafe. The proposed classes represent model-based comparative categories and must not be interpreted as validated diagnostic or structural-safety thresholds.

2.6 Sensitivity Analysis of Weighting Factors

Because the weighting factors influence the final ERI value, a sensitivity analysis is included. Three weighting scenarios are considered: the baseline model, an approximately equal-weighting model and a corrosion-current-oriented model.

Table 3. Weighting scenarios for ERI sensitivity analysis

Scenario	w_E	w_ρ	w_i	Interpretation
W1	0.30	0.25	0.45	Baseline model
W2	0.333	0.333	0.334	Approximately equal weighting
W3	0.20	0.20	0.60	Corrosion-current-oriented model

$$\Delta \text{ERI} = \text{ERI}_{\max} - \text{ERI}_{\min} \quad (9)$$

A small ΔERI indicates that the classification is relatively insensitive to the adopted weighting system, whereas a large value indicates stronger dependence on the selected weights. Particular attention is given to whether the scenario ranking $K0 > E1 > E2 > E3 > E4$ remains stable.

2.7 Evaluation of Conflicting Diagnostic Indicators

One advantage of the proposed multi-parameter model is its ability to address situations in which individual electrochemical indicators do not provide identical assessments. A structure may exhibit relatively negative reinforcement potential and moderate resistivity but low corrosion current density, or conversely, moderate potential together with low resistivity and elevated corrosion current density. The ERI therefore integrates electrochemical state, concrete conductivity and corrosion kinetics within a single comparative framework.

2.8 Statistical and Numerical Analysis

For future experimental implementation, each exposure condition should be represented by multiple specimens and the electrochemical parameters should be reported using mean values, standard deviations and coefficients of variation. In the present parametric numerical study, each exposure group is represented by a characteristic mean-state vector containing corrosion potential, resistivity and corrosion current density.

$$\text{CV} = (\text{SD} / \bar{X}) \times 100 \quad (10)$$

$$X_j = [E_{\text{corr},j}, \rho_j, i_{\text{corr},j}] \quad (11)$$

3 Results and Discussion

3.1 Parametric Electrochemical Response of the Exposure Groups

To evaluate the proposed Electrochemical Reliability Index, a representative parametric dataset was defined for the five exposure scenarios. The selected values describe a progressive transition from a favourable reference condition to a highly aggressive combined chloride-moisture-stray-current environment. The values represent a parametric numerical scenario and not experimentally measured data from a specific structure.

Table 4. Parametric electrochemical characteristics of the investigated exposure scenarios

Group	E_{corr} (mV)	ρ ($\text{k}\Omega \cdot \text{cm}$)	i_{corr} ($\mu\text{A}/\text{cm}^2$)
K0	-145	24.8	0.08
E1	-205	18.5	0.18
E2	-285	12.8	0.42
E3	-365	7.4	1.10
E4	-455	4.2	2.35

The adopted dataset indicates systematic deterioration of electrochemical conditions with increasing exposure severity. Corrosion potential becomes progressively more negative, concrete electrical resistivity decreases and corrosion current density increases from $0.08 \mu\text{A}/\text{cm}^2$ for K0 to $2.35 \mu\text{A}/\text{cm}^2$ for E4. This simultaneous evolution represents a transition from comparatively stable electrochemical conditions toward an increasingly active deterioration state.

The adopted values were selected within representative ranges reported in electrochemical corrosion literature and were arranged to form an internally consistent parametric progression from passive or low-activity conditions to highly active corrosion conditions.

3.2 Normalised Electrochemical Deterioration Parameters

Table 5. Normalised deterioration indicators

Group	R_E	R_ρ	R_i
K0	0.000	0.000	0.000
E1	0.194	0.306	0.044
E2	0.452	0.583	0.150
E3	0.710	0.845	0.449
E4	1.000	1.000	1.000

The deterioration components do not develop at identical rates. In E2, for example, $R_E = 0.452$ and $R_p = 0.583$, whereas $R_i = 0.150$. Thus, potential and resistivity already indicate substantial deterioration relative to the analysed range, while corrosion current density remains comparatively less developed. This is precisely the type of situation for which a multi-parameter model is useful.

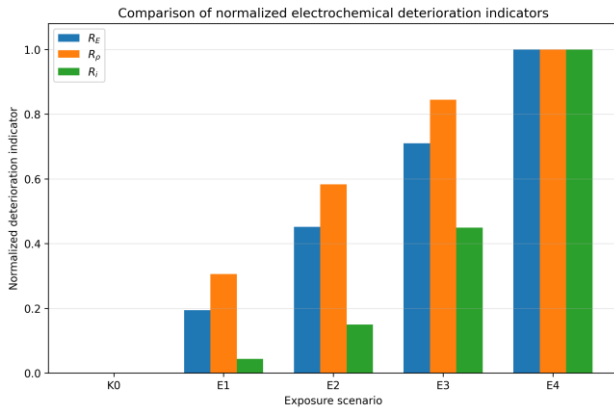


Figure 1. Comparison of normalised electrochemical deterioration indicators for K0-E4.

3.3 Electrochemical Deterioration Index and Reliability Index

Table 6. Calculated EDI and ERI values for the baseline weighting model

Group	EDI	ERI	Proposed reliability class
K0	0.000	1.000	Very high
E1	0.154	0.846	Very high
E2	0.349	0.651	High
E3	0.626	0.374	Low
E4	1.000	0.000	Very low

The ERI decreases systematically with increasing exposure severity: 1.000, 0.846, 0.651, 0.374 and 0.000 for K0 through E4, respectively. The ranking is therefore $K0 > E1 > E2 > E3 > E4$. The value $ERI = 0.000$ for E4 should not be interpreted as a literal probability of structural failure; it represents the lowest comparative electrochemical reliability state within the normalised dataset.

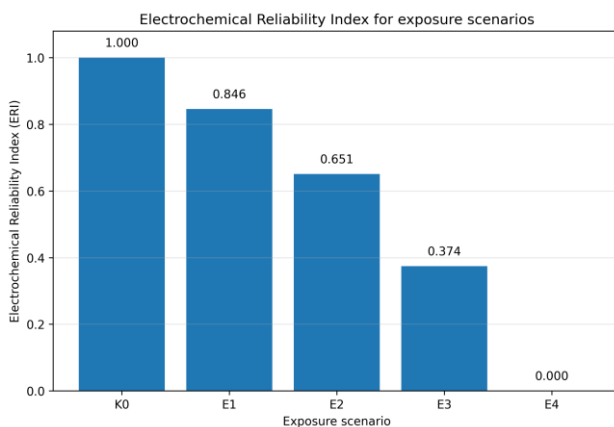


Figure 2. Electrochemical Reliability Index for the investigated exposure scenarios.

3.4 Influence of Stray Currents on Electrochemical Reliability

Comparison between E2 and E3 provides an indication of the contribution of simulated stray-current exposure. Corrosion current density increases from 0.42 to 1.10 $\mu\text{A}/\text{cm}^2$, corresponding to approximately 162%, while electrical resistivity decreases from 12.8 to 7.4 $\text{k}\Omega\cdot\text{cm}$ and corrosion potential shifts from -285 to -365 mV. The combined effect reduces ERI from 0.651 to 0.374, corresponding to a decrease of approximately 42.6% relative to E2. Within the proposed framework, stray-current exposure therefore represents a decisive transition from moderate electrochemical disturbance to significant deterioration risk.

3.5 Combined Influence of Moisture, Chlorides and Stray Currents

The E4 scenario represents simultaneous increased moisture, chloride contamination and stray-current influence. Compared with E3, corrosion current density rises from 1.10 to 2.35 $\mu\text{A}/\text{cm}^2$, resistivity decreases from 7.4 to 4.2 $\text{k}\Omega\cdot\text{cm}$ and potential shifts to -455 mV. The parametric results suggest that combined exposure produces substantially more severe electrochemical deterioration than stray-current exposure considered without the most aggressive environmental combination. From an engineering perspective, this combined condition should be regarded as particularly critical in reinforced concrete structures located near electrically influenced infrastructure.

3.6 Sensitivity Analysis of the Weighting Factors

Table 7. ERI values under different weighting scenarios

Group	ERI-W1	ERI-W2	ERI-W3	Δ ERI
K0	1.000	1.000	1.000	0.000
E1	0.846	0.819	0.874	0.055
E2	0.651	0.606	0.703	0.098
E3	0.374	0.332	0.420	0.087
E4	0.000	0.000	0.000	0.000

The most important result of the sensitivity analysis is that the ranking $K0 > E1 > E2 > E3 > E4$ remains unchanged for all three weighting systems. The greatest variation is obtained for E2, followed by E3, indicating that transitional exposure scenarios are more sensitive to weighting assumptions than the extreme states. Although the weights influence the magnitude of ERI, the overall comparative trend remains stable within the investigated scenario.

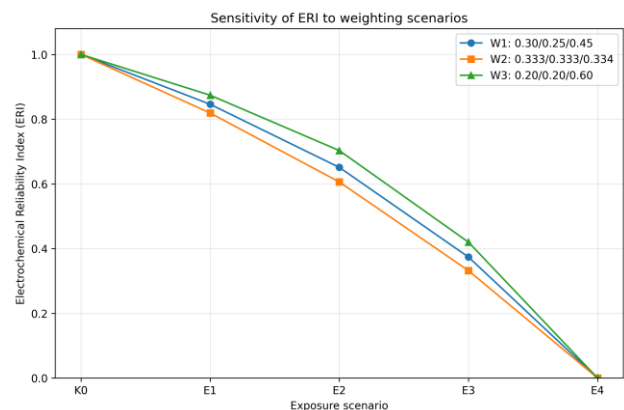


Figure 3. Sensitivity of ERI to the adopted weighting scenarios.

3.7 Identification of the Most Pronounced Modelled Transition

The most pronounced reliability reduction occurs between E2 and E3, where ERI decreases from 0.651 to 0.374. This identifies the

most pronounced transition within the adopted parametric scenario associated with simulated stray-current exposure. From a maintenance-management perspective, structures whose combined indicators place them near this transition may be priority candidates for more detailed investigation before severe corrosion propagation, cracking or cover spalling develop.

3.8 Multi-Parameter Versus Single-Parameter Assessment

The results demonstrate why a single electrochemical indicator may be insufficient. In E2, the normalised resistivity indicator suggests more pronounced deterioration than the corrosion-current indicator. If only one parameter were considered, the same condition could be classified very differently. By integrating all three indicators, $ERI = 0.651$ provides a balanced comparative assessment. The advantage of the proposed approach is therefore greatest in transitional conditions where individual parameters do not evolve synchronously.

3.9 Engineering Implications

The proposed ERI framework may support condition screening, risk classification, inspection prioritisation and maintenance planning. A high ERI would generally be associated with routine monitoring, whereas a substantial reduction in ERI could justify detailed electrochemical and structural investigation. ERI should not be interpreted independently as a structural safety index because structural reliability is ultimately influenced by reinforcement loss, cracking, bond degradation, geometry, material properties and applied loads. The proposed model is therefore best regarded as an electrochemical screening and comparative decision-support tool.

4 Conclusions

This study proposed a multi-parameter framework for the comparative assessment of electrochemical reliability of reinforced concrete structures exposed to different levels of electrical and environmental influence. Based on the adopted parametric dataset and numerical framework, the following conclusions can be drawn:

1. Increasing electrical and environmental aggressiveness produced a consistent deterioration of all analysed electrochemical parameters. Reinforcement potential became progressively more negative, electrical resistivity decreased and corrosion current density increased from the reference K0 condition toward the combined E4 scenario.
2. The proposed ERI successfully distinguished all five exposure scenarios. For the baseline weighting model, the calculated ERI values were 1.000 for K0, 0.846 for E1, 0.651 for E2, 0.374 for E3 and 0.000 for E4.
3. The transition from moderate electrical influence to simulated stray-current exposure represented the most pronounced reduction in comparative electrochemical reliability. The ERI decreased from 0.651 in E2 to 0.374 in E3, indicating that stray-current influence may represent a critical deterioration mechanism within the adopted parametric framework.
4. The combined action of moisture, chlorides and stray currents produced the least favourable electrochemical condition. The E4 scenario simultaneously exhibited the most negative reinforcement potential, the lowest concrete resistivity and the highest corrosion current density within the investigated dataset.
5. The multi-parameter approach demonstrated an important advantage over single-parameter assessment. In transitional scenarios such as E2, corrosion potential, resistivity and corrosion current density did not indicate identical deterioration severity. Their integration into ERI therefore provided a more balanced comparative interpretation.
6. Sensitivity analysis confirmed the stability of the exposure ranking. Although different weighting systems modified absolute ERI values, the ranking $K0 > E1 > E2 > E3 > E4$ remained unchanged for all analysed weighting scenarios.

7. Intermediate exposure conditions were more sensitive to the adopted weighting system than the extreme scenarios. The highest ERI variation was observed for E2, followed by E3, demonstrating that the selection of weighting coefficients is particularly important when individual indicators provide partially conflicting indications.

8. The proposed ERI should be interpreted as an electrochemical screening and comparative decision-support tool rather than as a direct structural safety index. It does not replace detailed structural inspection, chloride-content analysis, crack assessment, reinforcement-loss measurements or verification of load-bearing capacity.

The principal scientific contribution of the study is the development of an integrated framework that combines three complementary electrochemical indicators into a unified dimensionless reliability-oriented parameter. The model additionally incorporates sensitivity analysis of weighting coefficients, allowing the robustness of the final ranking to be evaluated.

A major limitation is that the analysed values represent a parametrically defined numerical scenario rather than experimentally measured data from a specific reinforced concrete structure. The proposed ERI classification limits and weighting coefficients should therefore be regarded as preliminary research assumptions requiring experimental validation.

Future research should include controlled laboratory exposure of reinforced concrete specimens to different levels of direct-current and stray-current influence, both with and without chloride contamination. Repeated measurements of reinforcement potential, concrete resistivity and corrosion current density should be combined with gravimetric reinforcement mass loss, chloride profiles, crack development and microstructural analysis. Such experimental data would enable calibration of the weighting coefficients and validation of the proposed ERI model for practical condition assessment and maintenance prioritisation.

Until experimental calibration is completed, the proposed ERI should be interpreted exclusively as a relative model-based electrochemical condition indicator.

References

1. Chen, Q., Zhang, Q., Zhu, T., Li, B., Liu, W., Yang, H., Xu, J., Jiang, Z., & Zhu, H. (2024). Coupling effect of concrete cracks and stray current on chloride-induced corrosion of rebar. *Construction and Building Materials*, 449, 138403. <https://doi.org/10.1016/j.conbuildmat.2024.138403>.
2. Liu, W., Xiao, J., Li, Y., Guo, Z., & Liu, H. (2025). Study on stray current corrosion behavior of rebars in cement mortar by XCT nondestructive testing method. *Construction and Building Materials*, 464, 140136. <https://doi.org/10.1016/j.conbuildmat.2025.140136>.
3. Chen, Y.-Q., Chen, M.-Y., Chen, R.-P., & Kang, X. (2023). Stray current induced ITZ effect on chloride transport in concrete. *Construction and Building Materials*, 409, 133759. <https://doi.org/10.1016/j.conbuildmat.2023.133759>.
4. ASTM International. (2022). ASTM C876-22b: Standard Test Method for Corrosion Potentials of Uncoated Reinforcing Steel in Concrete. ASTM International, West Conshohocken, PA.
5. RILEM TC 154-EMC. Test methods for on-site corrosion rate measurement of steel reinforcement in concrete by means of the polarization resistance method. RILEM Recommendation.
6. International Organization for Standardization. (2022). ISO 12696:2022 - Cathodic protection of steel in concrete. ISO, Geneva.

Mechanical Properties of Dissimilar Welded Joints Between Duplex Stainless Steel and Structural Steel

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Abstract: Dissimilar welding between duplex stainless steel and structural steel is increasingly used in the offshore, petrochemical and energy industries due to the combination of high corrosion resistance and good mechanical strength. This study presents a comparative investigation of two welding procedures for which they were used as filler metal: a BÖHLER Q G 309L-Si solid wire (GMAW 135 process), a FOX core 2209-T1 flux-cored wire (FCAW 136 process) and M21 shielding gas (82% Ar + 18% CO₂). The butt joint was performed between 10mm thick plates made of duplex stainless steel UNS S32205 and structural steel S355J2. During tensile testing, failure occurred in the base material, confirming the high strength and good quality of the welded joints. Charpy impact testing revealed high toughness in the weld area for the GMAW 135 process, while excellent toughness was obtained for the GMAW 136 process. Hardness measurements showed a more uniform hardness distribution for the flux-cored wire welds and macrostructural analysis confirmed full penetration of both welds.

Keywords: MECHANICAL PROPERTIES, DISSIMILAR WELDING, DUPLEX STAINLESS STEEL, CARBON STEEL

1. Introduction

Dissimilar welded joints are increasingly used in offshore structures, pressure vessels, petrochemical plants and energy systems, where the combination of corrosion-resistant materials and high-strength structural steels provides both economic and technical advantages [1-2], [3]. One of the most common combinations is duplex stainless steel UNS S32205 and structural steel S355J2. Duplex stainless steel UNS S32205 is a widely used engineering material characterized by a dual-phase microstructure consisting of approximately equal proportions of ferrite and austenite. This combination provides high mechanical strength, excellent resistance to stress corrosion cracking and superior corrosion resistance compared to conventional austenitic stainless steels [4-5]. In these steels, it is desired to maintain the phase proportion of approximately 40% to 60% ferrite, with the remainder being austenite, by adding austenite stabilizers, such as Ni, C, and Mn, in balance with ferritizing elements, such as Cr and Mo.

The effects of nitrogen on stainless steels are multiple, promoting both increased corrosion resistance (extension of the passive potential) and austenite content or mechanical strength. The microstructure in the heat-affected zone during welding is influenced by the thermal regime through the heat input in relation to the material thickness, the preheating temperature or the interlayer temperature. In other words, the maximum value of the exposure temperature controls the proportion of dissolved austenite and the diameter of the ferrite grains. The increase in the size of the ferritic grains has the effect of delaying the formation of austenite and, therefore, disturbing the phase equilibrium in the steel [6-8].

Structural steel S355J2 is commonly employed in welded constructions due to its good weldability, adequate mechanical properties and economic advantages. The combination of S32205 duplex stainless steel and S355J2 structural steel is frequently used in offshore, petrochemical and energy applications where both corrosion resistance and structural strength are required [2, 5-6].

The welding of these dissimilar materials presents significant metallurgical challenges because of differences in chemical composition, thermal expansion coefficient and phase transformation behaviour during welding thermal cycles [2], [5]. Improper control of welding parameters may lead to undesirable microstructural changes, residual stresses or deterioration of mechanical properties [1], [9].

The selection of filler material and welding process plays a crucial role in achieving sound dissimilar joints with adequate mechanical and metallurgical properties [9]. Gas Metal Arc Welding (GMAW) is widely employed in industrial applications because of its high productivity, good deposition rates and versatility [2].

Among the various GMAW variants, process 135 utilizes solid wire electrodes, while process FCAW employs flux-cored wire

consumables, each providing different thermal and metallurgical conditions during welding. Several studies have demonstrated that welding parameters, filler metal composition and heat input significantly influence the microstructure, toughness, hardness distribution and overall performance of duplex stainless-steel weldments [1],[5]. However, comparative data regarding the behaviour of dissimilar joints between S32205 duplex stainless steel and S355J2 structural steel produced using GMAW and FCAW processes remain limited.

The objective of this study is to perform a comparative evaluation of the mechanical and metallurgical behaviour of dissimilar welded joints produced using GMAW processes. The investigation has included non-destructive testing, tensile testing, bend testing, Charpy impact testing, Vickers hardness measurements and macrostructural examination to identify the most suitable welding procedure for industrial applications involving dissimilar materials.

2. Materials and Methods

2.1. Materials

In the experimental study two dissimilar welding joints have been performed between duplex stainless steel UNS S32205 and structural steel S355J2 plates, with 10 mm thickness.

The chemical composition of S32205 steel contains (wt.%): 0.03 C; 2 Mn; 1 Si; 21 – 23 Cr; 2.5 – 3.5 Mo; 4.5 – 6.5 Ni; 0.08 – 0.2 N. Based on this chemical composition, a PREN corrosion index value of 34 results, which is an average value in the duplex steel class. It has a tensile strength of 755 MPa, the yield strength of 582 MPa, Rockwell hardness HRC of 28 and elongation of 35%.

The chemical composition of S 355 J2 is (wt.%): 0.15 C; 1.38 Mn; 0.1 Si; 0.1Cr; 0.04Al; 0.1Ni. The main mechanical properties of this steel are: Tensile strength of 550 MPa, yield strength of 405 MPa, Brinell hardness of 170HB and elongation of 23%.

From the analysis of the property values of the steels that were welded, it is observed that problems can arise due to the diffusion of carbon from S 355 J2 towards the weld and the failure of the joint in the component made of this steel. The samples were machined for "V" butt –bevel shape, having an angle of 60°, depth of root face of 1.5 to 2mm, root gap of 2 to 4 mm (Fig. 1) [10]. Before welding, a layer of 309L-Si stainless steel was cladded on the joint area of the carbon steel part. The use of this filler material over the entire section of the joint required complex management of the rutile slag layer, a factor that decisively influenced the volumetric integrity of the weld, as observed during radiological control (Fig. 1b).

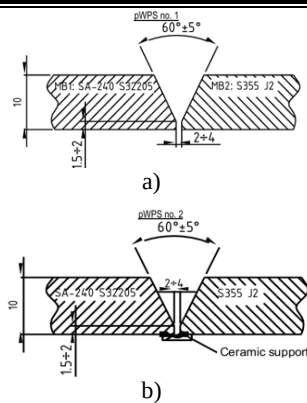


Fig. 1. Joint geometry and layers configuration for welded samples: a) GMAW 135 process; b) FCAW 136 process [9].

In both welding processes, shielding gas type M21 (18 %CO₂ and 82% Ar) was used. To prevent oxidation of the root weld of the sample welded with solid wire, root protection was performed using a separate cylinder with Argon (I1) shielding gas with a purity of 99.996%, that is the specific method applied in the case of stainless steels welding. To make the sample welded with tubular wire, the root was protected with a ceramic support. Two Gas Metal Arc Welding (GMAW) procedures were investigated in the study.

Welding process GMAW 135 has been performed using a solid wire electrode (BÖHLER Q G 309L-Si), while FCAW (Flux-Cored Arc Welding) 136 process utilized a flux-cored wire electrode (FOX core 2209-T1), both of them having 1.2mm diameter. BÖHLER Q G 309L-Si wire has an austenitic steel with the chemical composition of (wt.%): 0.02C; 0.9 Si; 1.6 Mn; 23.1 Cr; 0.1 Mo; 13.3 Ni; 0.1 Cu. FOX core 2209-T1 cored wire is projected for welding of duplex steels, with the chemical composition of (wt.%): 0.03C; 0.69 Si; 0.96 Mn; 23.25 Cr; 3.05Mo; 8.97 Ni; 0.09 Cu; 0.14 N.

2.2. Methods

Welding was performed with LINCOLN ELECTRIC SPEEDTEC 405 SP inverter multi-process equipment, equipped with advanced arc monitoring functions. The preliminary Welding Procedure Specification documentation (pWPS) was developed in accordance with the requirements of the standard SR EN ISO 15609-1[11].

The influence of the welding process on the mechanical behaviour, hardness distribution and structural integrity of the welded joints was subsequently evaluated through destructive and non-destructive testing methods [11-18].

The welding parameters (presented in Table 1) and the interlayer temperature were monitored during the execution of both welding procedures. Technological parameters such as the welding regime, the filler materials used, the type of shielding gas, as well as the thermal conditions for making the joint were defined for each technology. The temperature was monitored throughout the welding process to ensure that the maximum interlayer temperature of 150°C was not exceeded. To counteract the effect of thermal stresses and avoid angular deformations, the two plates were positioned and rigidly fixed on a metal alignment support (Fig. 2).

Table 1. Welding parameters

Layer	Current, A	Voltage, V	Travel speed, cm/min	Heat input, kJ/mm
Sample 1				
Root	110	16	24	0.40
L2	160	24	28	0.73
L3	180	26	30	0.80
Sample 2				
Root	130	22	25	0.69
L2	150	24	34	0.67
L3	170	25	22	0.98
L4	180	26	40	0.59

In this way, the start and completion of the weld could be done outside the welding area, thus avoiding the appearance of specific imperfections when starting and ending the electric arc burning process.

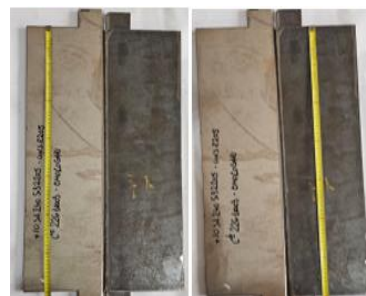


Fig. 2. The samples prepared for welding.

Upon visual inspection [12], both welded samples presented a satisfactory appearance after removal of welding spatter. The surface of the weld was clean, with regular geometry and uniform deposition of the filler material. When checking with penetrant liquids [13], it was observed that the welded samples had a compact surface, lacking relevant indications according to the acceptability criteria for quality level B (Fig. 3).

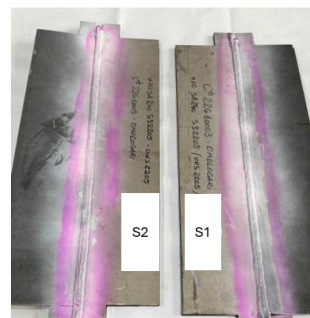


Fig. 3. Overview of welded samples after completion of liquid penetrant inspection.

Following the radiographic examination (RT), the solid wire welded sample did not present any unacceptable defects according to the criteria specified in the applicable standards [14].

The radiographic image obtained revealed a homogeneous structure of the weld, without major discontinuities such as cracks, inclusions or lack of penetration beyond the permitted limits, the sample being declared acceptable. In the case of the sample 2, welded with tubular wire, non-conforming internal discontinuities were highlighted, which required excavation and re-welding operations of the defective areas. The microstructure analysis was carried out in accordance with the requirements of the standard [15], using selective chemical attack to highlight the geometry and heat-affected zones. The Tensile tests and bending tests have been

performed using Universal tensile test machine OPTIMUM MB4P (12 t) [16] and bending tests have been performed using a hydraulic press (30 t) with bending rollers (diameter of 40mm), to verify ductility and inter-layer fusion [17]. Charpy impact tests were carried out at a temperature of -20 °C and the dimensions of the samples were 10x10x55mm, using a Wolpert PW 30/15 pendulum [18].

The measurement of hardness was performed by the Vickers method (HV0.5), according to the SR EN ISO 6507-1:2023 standard [19], using Shimadzu HMV 2T apparatus, for the evaluation of the local variation of mechanical properties in the characteristic areas of heterogeneous welded joints: base metal (S355 J2 and Duplex 2205), heat-affected zone (HAZ) and deposited metal. The measurements were performed using a force of 4.9 N (HV0.5) and a holding time of 10 s, according to the standardized methodology.

3. Results and discussion

Representative specimens were cut from the welded joints, processed according to international standards, for the following determinations:

between the joined materials.

Table 2. Comparative results of transverse tensile tests

Sample	Notch location	KV values, J			Average KV, J
Sample 1	Weld	122.5	100	112.5	112
	FL-CS	90	90	80	87
	FL-SS	122.5	92.5	147.5	121
Sample 2	Weld	72.5	75	62.5	70
	FL-CS	70	115	77.5	88
	FL-SS	182.5	122.5	147.5	151

FL-CS: fusion line from Carbon Steel; FL-SS: fusion line from Stainless Steel.

Table 3. Impact energy values (KV) obtained for samples of standard size

Sample	Width, mm	Thickness, mm	Surface, mm ²	Force, N	Rm, MPa	Failure zone	Result
T1.1	11.26	9.92	111.70	69.18	619.34	Base metal	Approved
T1.2	11.62	9.95	115.62	69.99	605.35	Base metal	Approved
T1.3	11.09	9.78	108.46	66.68	614.79	Base metal	Approved
T2.1	11.73	9.97	116.95	71.54	611.71	Base metal	Approved
T2.2	12.08	9.86	119.11	74.05	621.69	Base metal	Approved
T2.3	11.83	9.88	116.88	71.77	614.05	Base metal	Approved

Regarding the impact resistance test results, as shown in Table 3, it is observed that the fracture energy values are higher in the case of welded sample 2, especially in the fusion line with the stainless steel. However, the fracture energy values are lower in the welded interface with the structural steel. However, the lowest values of breaking energy were recorded in the weld made with tubular wire (sample 2), of maximum 75J, while in the sample welded with solid wire (sample 1) it was of minimum 87J.

The preparation of the specimens followed a rigorous protocol, using high-grain finishing equipment available in the laboratory to prevent alteration of the two-phase microstructure of the duplex steel. Macroscopic analysis of the fracture surfaces for both series of samples was carried out. As can be seen in the macroscopic analysis of sample 1 (Fig. 4a), it shows complete penetration and good fusion at both interfaces (duplex and carbon steel), from which it is found that it does not present macroscopic defects.

- 3 specimens from each sample were subjected to transverse tensile testing, to determine the ultimate strength (Rm) and tensile strength (Rp). The values obtained were targeted in the range of 470–630 MPa, to ensure compatibility with the strength limits of the base materials joined [4] (Table 2).
- 4 specimens taken from the samples for transverse bending tests: 2 specimens for root bending (TRBB) and 2 for face bending (TFBB).
- 3 sets of specimens for the impact bending tests (weld, duplex base material heat-affected zone and carbon base material heat-affected zone) (Table 3).
- one specimen from each welded sample for macroscopic examination (fig. 4).

In the case of Sample 1 (welded using solid wire electrode) most of the specimens showed lateral plastic deformations and a fibrous-looking fracture, suggesting high toughness.

For Sample 2 (welded using flux-cored wire electrode), the fracture surfaces indicated a mixed behaviour, highlighting the influence of the flux in the cored wire on the crystallinity of the weld metal.

In the case of bending tests, the good results are associated with the failure of the S355 J2 base material, without affecting the weld itself, thus highlighting a difference in mechanical behavior

Macroscopic analysis of sample 2 (Fig. 4b) highlights the stability of the cored wire welding process.



Fig. 4. Cross-sections through the welded samples. a) sample 1; b) sample 2.

A uniform and well-defined morphology of the weld beads is noted, as well as a metallurgical continuity without discontinuities (Fig. 5a). No internal defects were identified, but an axial rotational deformation occurred on the weld axis during the operation to remedy internal defects detected during the X-ray penetration test. In the case of sample welded using flux-cored wire electrode, the appearance of areas of partial mixing between the 2 alloys (filler

metal and S 355J2) as well as dispersed spherical inclusions is observed (Fig. 5b). The hardness distribution shows high values in the duplex base metal (331–333 HV0.5) and lower values in the S355 J2 carbon steel (212–214 HV0.5), corresponding to the differences in microstructure.

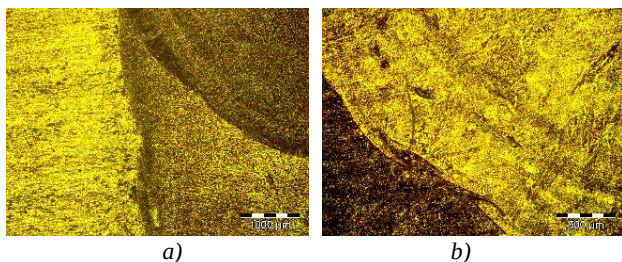


Fig. 5. Optical microstructure of cross section on fusion line. a) sample 1; b) sample 2.

In the Heat-Affected Zone (HAZ) of the S355 steel, an increase in hardness (265–270 HV0.5) is observed, determined by the microstructural transformations induced by the welding thermal cycle. The HAZ of the duplex steel shows high values (318–319 HV0.5), indicating the maintenance of the two-phase structure. The weld metal shows intermediate values (203–233 HV0.5), characteristic of a predominantly austenitic structure, which leads to a difference in hardness compared to the duplex steel. In the case of sample 2, the hardness values for the base metal remain similar to those of sample welded using solid wire electrode, but significant differences are observed in the deposited metal area.

The weld metal shows high values (351–355 HV0.5), close to those of duplex steel, indicating superior mechanical compatibility. This is determined using duplex filler material. The heat-affected zone of the carbon steel shows lower values (225 HV0.5), indicating a different influence of the thermal cycle. Sample welded using flux-cored wire electrode presents a more uniform distribution of hardness and a more gradual transition between zones, which indicates a superior mechanical compatibility. The results are consistent with the literature on the influence of the filler material on the mechanical properties of dissimilar welded joints [5–8].

4. Conclusion

The analysis of the mechanical test reports highlights that both samples meet the acceptance criteria for the tensile and impact tests, confirming good mechanical behavior of the dissimilar welded joints. For sample 1, all tensile specimens were accepted, and the rupture occurred in the base material, which indicates a superior resistance of the weld bead. In the case of bending tests, the “failure” results are associated with the failure of the S355 J2 base material, without affecting the weld itself, thus highlighting a difference in mechanical behavior between the joined materials. For sample 2, the results indicate a mixed behavior in bending, with specimens accepted on the weld face and rejected on the root, which highlights a different stress distribution and a more pronounced influence of technological parameters on the quality of the joint. The high fracture energy values (121 and 151 J in fusion line area) confirm an excellent toughness of the joint, especially at the interface with stainless steel, being in line with the microstructural observations on the metallurgical compatibility.

The experimental results highlight good compatibility near the fusion line with the duplex steel and a stable dendritic structure in the weld area. For sample welded using flux-cored wire electrode, the results indicate a mixed behavior in bending, with specimens accepted on the weld face and rejected on the root, which highlights a different stress distribution and a more pronounced influence of technological parameters on the quality of the joint.

The high fracture energy values (up to 150 J in the duplex area) confirm an excellent toughness of the joint, especially at the interface with stainless steel, being in line with the microstructural observations on the metallurgical compatibility.

The microstructure shows good compatibility with the fusion line at interface with duplex steel and a stable dendritic structure in the weld area. The Vickers hardness analysis highlights a differentiated distribution of mechanical properties between the characteristic areas of the joint, sample welded using flux-cored wire electrode presenting a higher homogeneity and hardness values closer to the duplex steel, which indicates a superior mechanical compatibility of the weld metal.

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References

- [1] Lippold, J.C., Kotecki, D.J., *Welding Metallurgy and Weldability of Stainless Steels*, Wiley-Interscience (2005).
- [2] Weman, K., *Welding Processes Handbook*, 2nd Edition, Woodhead Publishing (2012).
- [3] Kou, S., *Welding Metallurgy*, 2nd Edition, John Wiley & Sons, New York (2003).
- [4] J. Charles, Duplex Stainless Steels, A Review After DSS' 07 Held in Grado, *Revue de Métallurgie, Int. J. Metall.*, 105(3), p.155–171, <https://doi.org/10.1051/metal:2008028> (2008)
- [5] Besliu, M. M.; Voiculescu, I.; Solomon Gh., Effects of Dilution on Weld Overlays Realized with Flux-Cored Arc Welding (FCAW) Process Using 309LV Filler Metal on the S235JR Steel, *UPB, Sci. Bull. Series B-Chemistry and Materials Science*, Vol. 79, Issue: 1, p. 173-182 (2017).
- [6] Westin E. M., Putz A. et al., Solidification cracking in duplex stainless steel flux cored arc welds. Part 1— cracking in 30mm thick material welded under high restraint, *Welding in the World* 66:2405–2423(2022).
- [7] Moteslakker A., Danaee I., Microstructure and Corrosion Resistance of Dissimilar Weld-Joints between Duplex Stainless Steel 2205 and Austenitic Stainless Steel 316L, *Journal of Materials Science & Technology* 32 p. 282–290 (2016).
- [8] Aksascki Caetano G. et al. Duplex Stainless Steels – Mechanical Properties Study of a Duplex Stainless-Steel Weld Using Physical Simulation and Work Hardening Models, *Journal of Materials Engineering and Performance*, № 1, p. 113-127 (2021).
- [9] Voiculescu, I., Geantă, V., Vasile, I.M., Ștefănoiu, R., Tonoiu, M., Characterization of weld deposits using as filler metal a high entropy alloy, *Journal of Optoelectronics and Advanced Materials*, Vol. 15, No. 7-8, p.650 – 654 (2013).
- [10] BS 499-1:2009. Welding terms and symbols. Glossary for welding, brazing and thermal cutting (British Standard).
- [11] SR EN ISO 15614-1:2017+A1:2019, Specification and qualification of welding procedures for metallic materials – Welding procedure test.
- [12] SR EN ISO 17637:2017, non-destructive testing of welds – Visual testing of fusion-welded joints.
- [13] SR EN ISO 3452-1:2021, non-destructive testing – Penetrant testing – Part 1: General principles
- [14] SR EN ISO 17636-1:2022, non-destructive testing of welds – Radiographic testing – Part 1.
- [15] SR EN ISO 17639:2022, Destructive tests on welds in metallic materials – Macroscopic and microscopic examination of welds.
- [16] SR EN ISO 4136:2022, Destructive tests on welds in metallic materials – Transverse tensile test.
- [17] SR EN ISO 5173:2023, Destructive tests on welds in metallic materials – Bend tests.
- [18] SR EN ISO 148-1:2017, Metallic materials – Charpy pendulum impact test – Part 1: Test method.
- [19] SR EN ISO 6507-1:2023, Metallic materials – Vickers hardness test – Part 1: Test method.

Main casting defects at the interface between a cast iron ring insert and an al-si piston

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Abstract: The study identifies the main casting defects at the interface between a cast iron ring insert and an Al-Si piston and determines their principal causes. Industrial investigations of 1000 pistons showed that the Al-Fin process increases Fe content in the AlSi9 alfining alloy, promoting long plate-like β -Al₅FeSi phases and porosity in the joint region. Gas contamination by oxides and hydrogen further weakens the bond. The addition of approximately 0.05 wt.% Mn and intermediate re-refining of the AlSi9 alloy were found to improve the microstructure and reduce porosity, thereby increasing the quality of the insert-piston joint.

KEYWORDS: ALUMINIUM PISTONS, CASTING DEFECTS, IRON-RICH PHASES, POROSITY, AL-FIN PROCESS

1. Introduction

Depending on the type of internal combustion engine, different piston designs are used, consisting of multiple components. One of these is the ring zone, which accommodates the piston rings (usually 1 to 4 in two-stroke engines and 2 to 4 in four-stroke engines). Their functions include sealing the gases generated by combustion of the fuel-air mixture and preventing them from entering the lower part of the piston and the crankcase. To achieve this, the rings must conform to the shape of the cylinder bore over their entire diameter and width [1, 2].

To reinforce the piston-ring operating zone and dissipate heat from the upper part of the piston, the rings are seated in a cast iron insert in which a groove is machined for the appropriate ring. To obtain a sound bond between the Al-Si piston alloy and the cast iron ring inserts, the inserts are subjected to the so-called Al-Fin (alfining) process. This process consists of briefly immersing the insert in molten AlSi9 alloy in order to form a diffusion layer [3, 4]. After cooling, the inserts are placed in a permanent mould and overcast with the piston alloy. Unfortunately, the Al-Fin process causes the iron content and the level of other contaminants in the AlSi9 alloy to increase. These promote casting defects at the insert-piston interface, thereby increasing the proportion of defective products. Until recently, pistons with inserts were used only in heavily loaded diesel engines operating at combustion pressures of 2.5–3.5 MPa, compared with 1.0–1.5 MPa for gasoline engines. Currently, piston inserts are used in all types of internal combustion engines [5, 6]; therefore, identification of defects at the insert-piston interface is of major importance.

2. Prerequisites and means for solving the problem

The aim of the study was to identify the most important casting defects at the diffusion-bonded interface between a cast iron ring insert and a silumin piston casting, and to determine the principal causes of these defects and possible methods of limiting their occurrence.

The defects were identified using pistons for a 1400 cm³ gasoline engine (representing the largest share of orders), based on a population of 1000 randomly selected pistons.

The scope of the study included:

- identification of the main casting defects at the insert-piston interface,
- microstructural examination of the insert-piston joint region,
- identification of potential causes of insufficient adhesion between the insert and the piston,
- proposal of corrective solutions and analysis of their feasibility under industrial conditions.

2.1. Research problem

Statistical data from the Quality Control Department of Federal-Mogul Gorzyce sp. z o.o. indicate that casting defects, particularly those occurring at the insert-piston interface, constitute the most important group of causes of piston rejection. These nonconformities are detected only at the end of the manufacturing process during final piston quality inspection, which generates

additional losses of energy and time and reduces production-process efficiency.

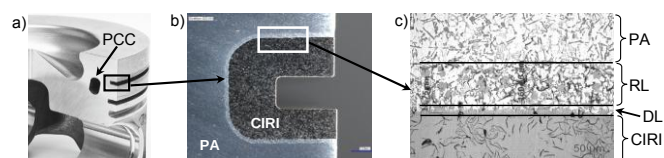
Preliminary investigations showed that these defects result from an improper insert-piston bond within the remelting and diffusion layers. They occur, among other reasons, when the permissible iron content in the AlSi9 alloy is exceeded as a result of:

- diffusion of iron atoms from the cast iron insert into the alfining alloy,
- accidental dropping of an insert into the molten alloy,
- lack of control of the iron content in the alloy during alfining, and
- excessive gaseous contamination of the AlSi9 alloy.

Reducing the formation of defects will lower costs, reduce machine utilisation and material consumption in subsequent production stages (machining and surface treatment of pistons). It is therefore essential to identify the causes of defect formation and implement corrective actions.

2.2. Materials and research methodology

The subject of the investigation was casting defects occurring at the interface between the insert and a gasoline-engine piston cast from AlSi11Cu4Ni3Mg alloy (Fig. 1). Identification of casting defects at the insert-piston interface and assessment of insert adhesion to the piston were carried out using the ultrasonic and penetrant testing methods employed at Federal-Mogul Gorzyce.



where:

- PCC – piston cooling channel,
- PA – AlSi11Cu4Ni3Mg piston alloy,
- RL – remelting layer,
- DL – diffusion layer,
- CIRI – cast iron ring insert.

Fig. 1. Object of the study: (a) fragment of a gasoline-engine piston; (b) insert-piston interface region; (c) correct microstructure of the insert-piston joint (property of Federal-Mogul Gorzyce sp. z o.o.).

According to the Federal-Mogul guidelines (standard TS1E-010-020), the bond between a ring insert made of Ni-Resist cast iron EN-GJLA-XNiCuCr15-6-2 according to TS1E-012-006 (DIN EN 13835:2012) and the piston casting around its circumference is considered unacceptable if at least one of the following conditions is met:

- the RL thickness is less than 70 μ m or greater than 400 μ m,
- the DL thickness is less than 5 μ m or greater than 40 μ m,
- the fraction of iron-rich phases in the RL exceeds 10% of the insert circumference,
- the presence of gaseous contamination, cracks, porosity and other defects is unacceptable.

The pistons were examined ultrasonically over the entire circumference of the insert while it was rotated about its own axis. Defects were examined on the upper and lower surfaces of the insert. One hundred percent of the products were subjected to quality-control testing of the insert-piston joint.

3. Results and discussion

Based on the investigations, the main causes of casting defects at the interface between the insert and the piston casting were found to be:

- an Fe content exceeding 0.5 wt.% in the AlSi9 alloy, resulting in the formation of iron-rich phases with an unfavourable morphology,
- gas contamination of the AlSi9 alloy, mainly by hydrides and oxides, causing porosity, and
- other casting defects between the insert and the piston cooling channel (PCC – Fig. 1).

Examples of microstructures of defective insert-piston joints containing unfavourable iron-rich phases are shown in Fig. 2; defects caused by excessive porosity are shown in Fig. 3, whereas other defects, mainly delaminations, are presented in Fig. 4.

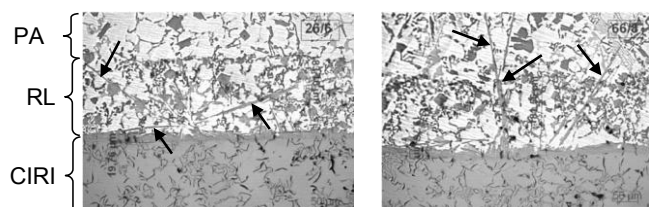


Fig. 2. Microstructures of an improper insert-piston joint containing iron-rich phases in the remelting-layer region (property of Federal-Mogul Gorzyce sp. z o.o.).

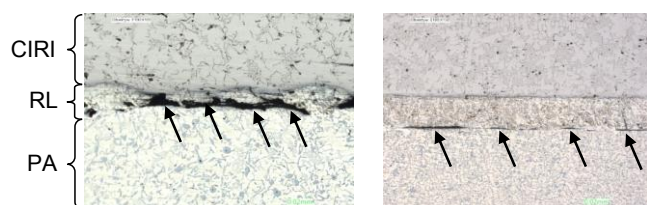


Fig. 3. Microstructures of an improper insert-piston joint showing porosity in the remelting-layer region (property of Federal-Mogul Gorzyce sp. z o.o.).



Fig. 4. Macrostructures of an improper insert-piston joint: (a) shrinkage cavities between the insert (CIRI) and the piston cooling channel (PCC); (b) porosity around the insert (property of Federal-Mogul Gorzyce sp. z o.o.).

3.1. Preventive measures

Microstructural analysis showed that the iron-rich phases present in the remelting layer are Al_5FeSi (the so-called β -Fe phase). To reduce their adverse effect, manganese was added to the AlSi9 alloy in the form of an AlMn50 master alloy (approximately 0.05 wt.% Mn). The literature analysis [7–9] indicates that manganese has a beneficial effect on the plate-like precipitates of the β -Fe phase. It was also found that after 3–4 h of alfining the inserts, the AlSi9 alloy should be subjected again to a refining treatment, for example with argon, after which the alfining process can be continued for a further 3–4 h. Examples of insert-piston joint microstructures after

the addition of manganese to the AlSi9 alloy and subsequent re-refining are shown in Fig. 5.

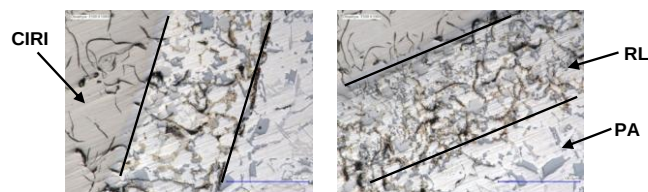


Fig. 5. Microstructures of a correct insert-piston joint after manganese addition and a repeated refining treatment (property of Federal-Mogul Gorzyce sp. z o.o.).

3.2. Discussion

The study was aimed at identifying the principal casting defects in the joint region between a cast iron ring insert and a piston casting made of AlSi11Cu4Ni3Mg alloy. Defects were identified on the basis of microstructural examinations of a population of 1000 randomly selected pistons for a 1400 cm³ gasoline engine. The investigations were carried out under industrial conditions at the Federal-Mogul Gorzyce sp. z o.o. foundry in Gorzyce. The investigations and observations showed that, during piston production, the most problematic operation is the Al-Fin treatment of the ring inserts. During this process, iron atoms from the cast iron inserts diffuse into the AlSi9 alloy, increasing its iron content. Consequently, long (500–800 μm), plate-like Al_5FeSi precipitates crystallise in the joint region between the cast iron ring insert and the piston casting (Fig. 2). These phases cause the insert-piston joint to become unacceptable from a quality standpoint, resulting in a higher scrap rate and associated losses.

Another problem during the Al-Fin treatment of ring inserts is that the process is carried out in open furnaces, which considerably increases the amount of non-metallic inclusions in the AlSi9 alloy, mainly oxides and hydrogen. This results in increased porosity in the insert-piston joint region (Fig. 3). The presence of pores weakens the material, first generating local stresses and then microcracks, which develop into cracks during piston operation. This occurs because long, needle-like β - Al_5FeSi precipitates whose major dimension is oriented perpendicular to the applied load undergo decohesion, whereas β - Al_5FeSi phases aligned parallel to the load crack along their arms and fragment [10]. These factors significantly reduce fatigue strength under long-term loading (more than 10^6 cycles), which is an important parameter for piston castings and contributes to a reduction in the strength of the cast iron ring–Al–Si piston joint.

Porosity between long β - Al_5FeSi phases reduces feeding capability by restricting the flow of liquid alloy between the dendrite arms of the $\alpha(\text{Al})$ solid solution. Such regions act as sites for microcrack initiation, reducing fatigue and static strength, impact toughness and ductility as expressed by elongation. In addition, the size of the needle-like β - Al_5FeSi precipitates, and particularly their highly unfavourable morphology, makes piston machining more difficult. Under the pressure exerted by the turning tool, β -Fe phase precipitates further reduce the durability of the insert-piston joint. Other casting defects were also observed (incorrect thickness of the remelting and diffusion layers, delaminations and shrinkage cavities – Fig. 4); however, their share was small.

To prevent these defects, two improvement measures were proposed:

- addition of manganese to the AlSi9 alloy, and
- an intermediate re-refining treatment of the AlSi9 alloy.

Microstructural examination of the AlSi9 alloy after manganese addition showed that, in addition to a small number of β - Al_5FeSi precipitates, the α - $\text{Al}_{15}(\text{Fe},\text{Mn})_3\text{Si}_2$ phase was present. Its morphology was dendritic or of the so-called “Chinese-script” type. The investigations also showed that an additional refining treatment of the AlSi9 alloy reduces the amount of gaseous contamination. This directly decreases porosity in the interface region between the

cast iron ring insert and the piston, thereby enabling a sound joint to be obtained (Fig. 5).

It should be emphasised that, in addition to the proposed measures aimed at improving the bond between cast iron ring inserts and the piston, further research should be continued, particularly with regard to further reducing iron contamination and non-metallic inclusions, especially oxides and hydrides.

4. Conclusion

Based on the investigations and analysis of the results, the following conclusions were formulated:

1. The process that generates the largest number of casting defects is the Al-Fin treatment of ring inserts. This process requires special supervision.
2. An excessive amount of morphologically unfavourable β -Al₅FeSi phases was identified in the AlSi9 alloy. Their size and fraction increase with increasing alfining time as the iron content rises. The presence of these phases is one of the principal causes of an unacceptable-quality bond between the insert and the piston.
3. Brittle β -Fe phases block interdendritic channels and impede the flow of liquid alloy, causing shrinkage porosity and shrinkage voids in the insert-piston interface region. Their presence increases the brittleness of the joint, while the resulting porosity produces numerous casting defects at the ring insert-piston interface.
4. Manganese has a beneficial effect on modification of the unfavourable β -Al₅FeSi phase morphology. Its action consists in 'binding' iron and promoting crystallisation of the Al₁₅(Fe,Mn)₃Si₂ phase. The addition of manganese to the AlSi9 alloy changed the morphology of the β -Fe phases into α -(Fe,Mn) phases.
5. Microstructural examination of the insert-piston joint region revealed the presence of gas pores. Their fraction can be reduced, for example, by introducing an intermediate alloy degassing treatment. Further investigations in this area should be continued.

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5. References

- [1] Kammer C.: Aluminium Handbook. Vol. 1: Fundamentals and Materials. Beuth Verlag GmbH, 2011, (e-book), <https://booksplusebooks.com>.
- [2] Jason Rowe: Advanced Materials in Automotive Engineering. Woodhead 2021.
- [3] Pietrowski S.: Structure of the Al-Fin layer on grey cast iron. Archives of Foundry, 2004, 251-266.
- [4] Czerepak M., Piątkowski J.: Crystallization of AlSi9 alloy designed for the alfin processing of ring supports in the engine pistons. Archives of Foundry Engineering 20, 2020.
- [5] Baldan R., Malavazi J., Cauto A.: Microstructure and mechanical behavior of Al9Si0.8Fe alloy with different Mn contents. Materials Science and Technology, 2017.
- [6] Crolla David A.: Automotive Engineering. Powertrain, Chassis System and Vehicle Body. Elsevier 2009, (e-book), <http://www.elsevierdirect.com/9781856175777>.
- [7] Shabestari S.G.: The Effect of iron and manganese on the formation of intermetallic compounds in Al-Si alloys. Materials Science and Engineering A, 383, 2004, 289-298.
- [8] Xinjin Cao and Campbell J.: Morphology of β -Al₅FeSi phase in Al-Si cast alloys. Materials Transaction, vol.47, No.5, 2006, 1303-1312.
- [9] Taylor, J.A.: Iron-containing intermetallic phases in Al-Si based casting alloys. Procedia Materials Science, vol.1, 2012, 19-33. DOI: 10.1016/j.mspro.2012.06.004.
- [10] Campbell J.: A hypothesis for cast iron microstructure. Metallurgical and Materials Transactions B, 2009 Research Gate. DOI: 10.1007/s11663-009-9289-0.