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IDENTIFICATION OF PHASE PERCOLATION IN BAINITIC STRUCTURES

IDENTYFIKACJA PERKOLACJI FAZ W STRUKTURACH BAINITYCZNYCH

Summary: The aim of this study was to identify and study the phenomenon of phase percolation in bainite structures. SEM microscopic tests were carried out on bainitized iron alloy samples, in the FEM tests austenitic SS316 steel was used as the filled phase. Physical models, which were produced in the form of cubes and tensile samples, were made in FDM technology on the Original Prusa Mini+ printer from ASA filament (modified ABS copolymer). The static tensile test was performed on a LabTest 5.20 SP1 testing machine at a tensile speed of 10mm/min. Complex states of stress in the analyzed systems were identified, which prove the complexity of the phenomenon in multiphase materials. The obtained results were correlated, which allowed to indicate that the phenomenon of percolation indeed affects the mechanical properties of multiphase materials and incremental techniques can be used to analyze the level of this impact.

Keywords: phase percolation, bainite structures, mechanical properties of multiphase materials, incremental techniques

Streszczenie: Celem pracy było rozpoznanie i zbadanie zjawiska perkolacji fazowej w strukturach bainitu. Badania mikroskopowe SEM przeprowadzono na bainityzowanych próbkach stopu żelaza, w badaniach MES jako fazę wypełnioną zastosowano stal austenityczną SS316. Modele fizyczne, które wykonano w postaci kostek i próbek rozciągliwych, wykonano w technologii FDM na drukarce Original Prusa Mini+ z filamentu ASA (modyfikowany kopolimer ABS). Statyczną próbę rozciągania przeprowadzono na maszynie wytrzymałościowej LabTest 5.20 SP1 przy prędkości rozciągania 10 mm/min. Zidentyfikowano złożone stany naprężeń w analizowanych układach, które świadczą o złożoności zjawiska w materiałach wielofazowych. Uzyskane wyniki skorelowano, co pozwoliło wskazać, że zjawisko perkolacji rzeczywiście wpływa na właściwości mechaniczne materiałów wielofazowych, a techniki przyrostowe mogą być wykorzystane do analizy poziomu tego oddziaływania..

Słowa kluczowe: perkolacja fazowa, struktury bainitu, właściwości mechaniczne materiałów wielofazowych, techniki przyrostowe

Introduction

Percolation in materials is most often described as a process in which a liquid or gas diffuses through a network of interconnections in a porous material. This is an important phenomenon in fields such as materials science, chemistry, chemical engineering and environmental science. There are several methods of recognizing percolation in materials, depending on its type and expected results. Among commonly used methods distinguished are:

- Porosity analysis: The use of microscopic techniques such as optical or electron microscopy allows examine the structure of the material and identify porosity. Image analysis can provide information on pore size, shape and distribution, which can help identify potential percolation paths.
- Electrical conductivity measurement: This technique is particularly useful for conductive materials such as metal or conductive polymers. Electrical conductivity measuring devices such as multimeters or impedance analyzers can be used to monitor changes in conductivity as liquid or gas penetrates.
- Permeability tests: To assess percolation in porous materials such as rocks, soils or filter membranes, permeability tests can be performed. For example, a permeametric test can measure the flow of liquid through a sample of the material and provide information about percolation.
- Computer simulations: recognizing percolations in multiphase materials can be difficult with traditional methods, so computer simulations are used in many cases. Numerical models and simulations can help analyze and visualize the percolation process in the material.

It should be noted that the appropriate percolation detection method depends on the specific material and the purpose of the test.

Percolation is characteristic of porous or non-metallic materials with networks of connections between the pores. In metal structures, such as monolithic metal blocks or sheet metals, there are typically no pore networks through which liquid or gas can penetrate. If there are porous structures in the metal structure, e.g. in metal composites or metal coatings on porous substrates, there is a possibility of percolation through these porous structures. In such cases, percolation may refer to the

penetration of a liquid or gas through the pore network connections of those porous materials that are present within the metal structure.

In the literature, however, the term "phase percolation" can be found, which refers to the phenomenon of percolation occurring between different phases or components in a composite material. In composite materials, two or more distinct phases are combined to form a material with desired properties. These phases can be different materials, such as polymers and fillers, or different states of matter, such as solid and liquid.

Phase percolation occurs when a continuous path or network forms between the phases, allowing physical or chemical properties to be transferred throughout the composite material. This percolation network can allow different quantities to be transported, such as electrical conductivity, thermal conductivity or fluid flow, depending on the specific properties of the phases involved.

The percolation threshold is an important concept in phase percolation. It represents the critical volume or concentration at which a single-phase interconnected lattice begins to form in a composite material. Below the percolation threshold, the phases remain isolated and do not significantly affect the overall material properties. Once the percolation threshold is reached, there is an abrupt change in the behavior of the material as the interconnected network of the percolating phase begins to dominate the properties of the composite. By controlling the percolation behavior, composite properties can be optimized and advanced materials with properties tailored to specific applications can be developed [1].

Influence of percolation on mechanical properties

Percolation can have a significant effect on the mechanical properties of multiphase materials. When a composite material percolates, the formation of an interconnected network or path between the different phases can affect its overall mechanical behavior in several ways:

- Strength and stiffness: The presence of percolation paths can increase the overall strength and stiffness of the composite material. The material's load capacity increases as stresses are transferred more efficiently between phases through the percolation network. This can result in improved mechanical properties such as increased tensile strength, compressive strength and Young's modulus.

- Strength and Fracture Resistance: Percolation can also improve the strength and fracture toughness of composite materials. The interconnected network provides stress redistribution and energy dissipation paths, preventing crack propagation and increasing the material's ability to absorb and withstand applied loads without catastrophic failure.

- Elastic behavior: The presence of percolation paths can affect the elastic behavior of the composite material. Percolation phases can contribute to the overall elastic response by affecting parameters such as elastic modulus and Poisson's ratio. The stiffness and deformation characteristics of the composite may be influenced by changes in the material microstructure caused by percolation.

- Anisotropy: Percolation can introduce anisotropic behavior in composite materials, meaning that their mechanical properties can change depending on the direction. The orientation and placement of the percolation paths can affect the material's response to different types of loads, resulting in different mechanical properties along different axes or directions.

It should be noted that the extent and nature of the percolation, including the percolation threshold, the morphology of the percolation network, and the properties of the phases involved, can play a role in determining the specific effect on mechanical properties. Therefore, optimizing the percolation behavior is a key factor in designing composite materials with desirable mechanical properties for various applications. Therefore, scientific work is undertaken aimed at building physical models testing various types of phase percolation in search of the best individual properties of the material. This paper focuses on austenite percolation in bainitic structures.

Austenite percolation in polycrystalline structures

In the context of solid structures, especially in the presence of multiple phases, the term "percolation" can be used to describe the spatial connectivity and propagation of a particular phase in a microstructure (Fig. 1).
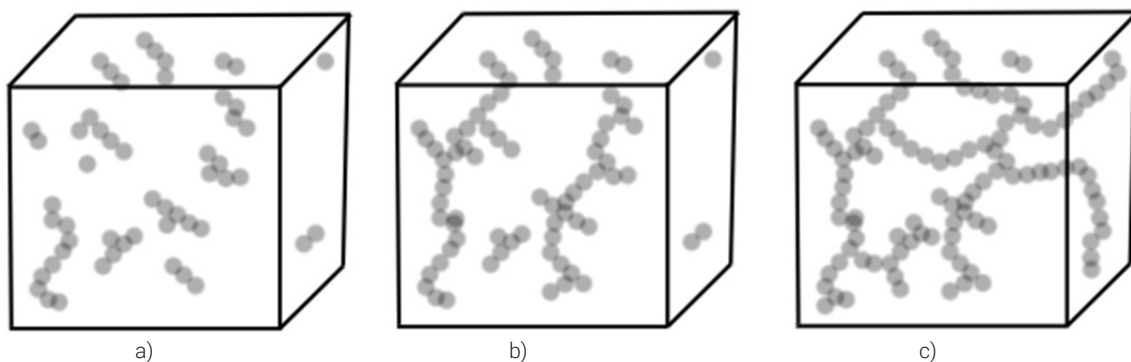

a)　　　　　　b)　　　　　　c)

Fig. 1 Schematic illustration of critical volume fraction in the percolative network of spherical inclusions in the random distribution: a) without percolation; b) critical volume fraction percolative network; c) percolative network cluster
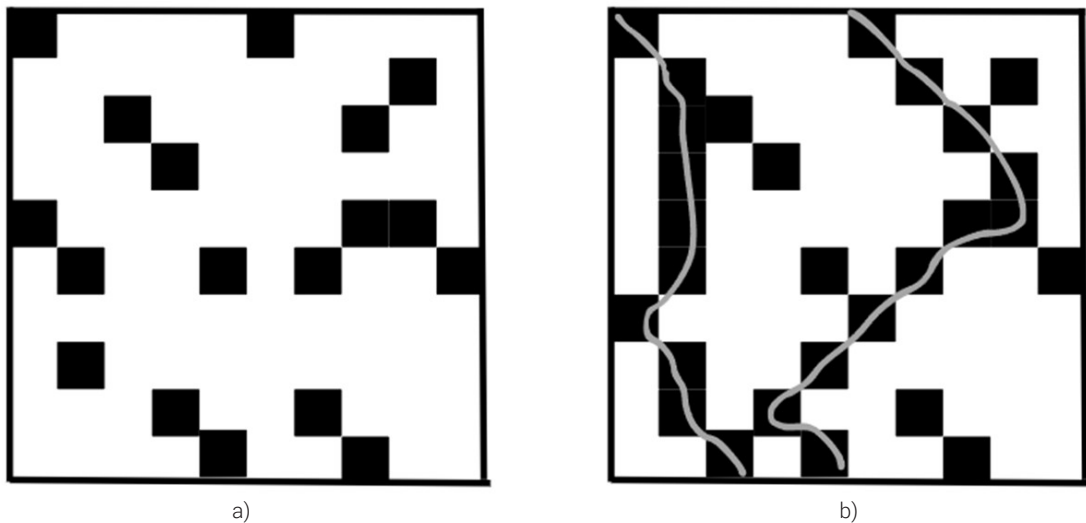
Fig. 2. Percolation illustration: a) black phase is below this threshold; b) black phase contributes above the percolation threshold

In the case of austenite, percolation may occur in solid structures containing a mixture of austenite and other phases, such as ferrite or cementite. Percolation refers to the phenomenon in which the austenite phase forms a continuous path throughout the material, connecting multiple regions or grains [2]. This interconnected path allows specific material properties to be affected, e.g. hydrogen diffusion in bainitic steel [2] or yield strength in duplex steel [3].

The percolation of austenite in solid structures depends on various factors, including temperature, time, composition, and the presence of nucleation sites or defects. The kinetics and extent of percolation can be affected by atomic diffusion, growth of new phase regions, and movement of phase boundaries. However, a method of determining it must be found.

Experimental techniques such as microscopy, diffraction and thermal analysis can be used to observe and analyze austenite percolation in solid structures. These techniques provide insight into the evolution of the microstructure and connectivity of the austenite phase. Physical modeling techniques can also be used for this analysis, but this requires a high level of understanding of the mechanisms that create the percolating structure.

Percolation of austenite in bainite

The transformation of austenite into bainite occurs through the nucleation and growth of a new phase (ferrite) that occurs within the existing austenite grains (Fig. 3). In the case of modern nanobainitic steels, the transformation results in a microstructure with a unique morphology of fine, lamellar ferrite in an austenite matrix with little or no carbide precipitates. The extent and kinetics of nanobainite formation depend on such factors as temperature, alloy composition and time [4]. Recently, the best property indicators have been documented when only ferrite and austenite (Fig. 3b) are present in the microstructure - such a structure model was adopted for the analysis [4].
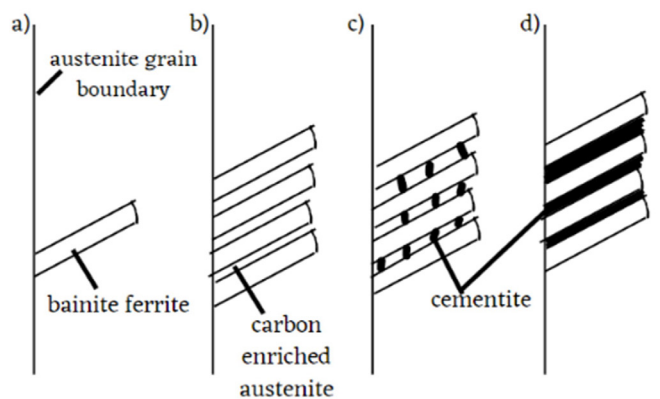

Fig. 3. Scheme of the formation of upper bainite a); b); c); d) successive stages of transformation
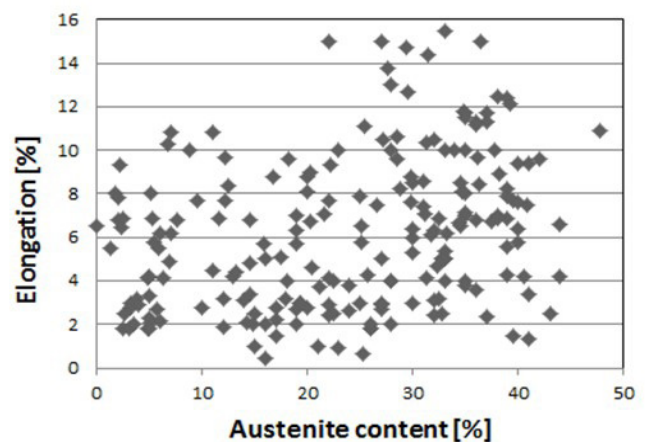

Fig. 4. Dependence of elongation on the share of austenite in the structure of austempered ductile iron

In the case of heat-treated iron alloys, e.g. bainitic steel or austempered ductile iron, it is known that the increasing volume fraction of austenite changes the mechanical properties, but this change is difficult to justify with simple dependencies

[5]. Austenite is a plastic phase, so it would be natural for the material to become more plastic as the volume fraction of this phase increases. However, the dependence of the elongation determined in the static tensile test as a function of the austenite share shows that additional indicators are needed to describe the spatial structure of the material (Fig. 4). In addition, it was proven that below 10% of austenite, brittle cracking of the material occurs due to the breaking of austenite percolation chains [5,8].

This observation can be understood if one assumes that failure occurs when the austenite, which is the hardest of all phases present, becomes geometrically isolated, i.e. loses its percolation, leading to crack formation [6]. Garboczi et al. developed a numerical model for the percolation threshold when freely overlapping objects (ellipsoids) are in the matrix [7]. Since austenite is roughly divided by lamellar ferrite, it can be represented by flattened ellipsoids with an aspect ratio r of about 1/10 to 1/100. It was then found that the percolation threshold is $p_c \sim 1.27r$, i.e. $0.127 \geq p_c \geq 0.0127$. This is consistent with the observation that tensile failure occurs when $V_\gamma \sim 0.1$. It was therefore found that the austenite percolation must occur in order to obtain specific mechanical properties of the structure of the austenitic-ferritic mixture. It was indicated that austenite content of at least 10% results in the continuity of the austenite path. However, the question remains how to visualize it and extract it from the system in order to study the best geometric features of the system.

Creating a model of austenitic-ferritic structure in carbide-free bainite

In order to simplify the task, the creation of a virtual model of a single elementary cell of the austenitic-ferritic structure was started with the assumption that one time and temperature state of the material is considered, in which the heat treatment process of a specific type of iron alloy ends. Growing ferrite plates nucleate and then fill individual grains of austenite. It was therefore assumed that this austenite grain with a specific number and morphology of ferrite plates would represent a single cell of the modeled structure.

The assessment and analysis of the microstructure of the described mixture of phases shows that ferrite plates nucleate at the boundaries of austenite grains (Fig. 5) and that they have the form of plates visible not only by reconstruction, but also after deep etching and observation in an electron microscope (Fig. 5). For smaller microscopic magnifications, the privileged directions of growth of this structure are also visible (Fig. 5).

Taking into consideration the above assumptions, a simplified model of the structure of a single grain in the structure of carbide-free bainite, i.e. a mixture of lamellar ferrite and austenite, was developed in the SolidWorks program (Fig. 6) This model was called the basic cell. A cross-section through such a cell in non-obvious planes indicates a complex spatial system of two different materials interpenetrating each other.
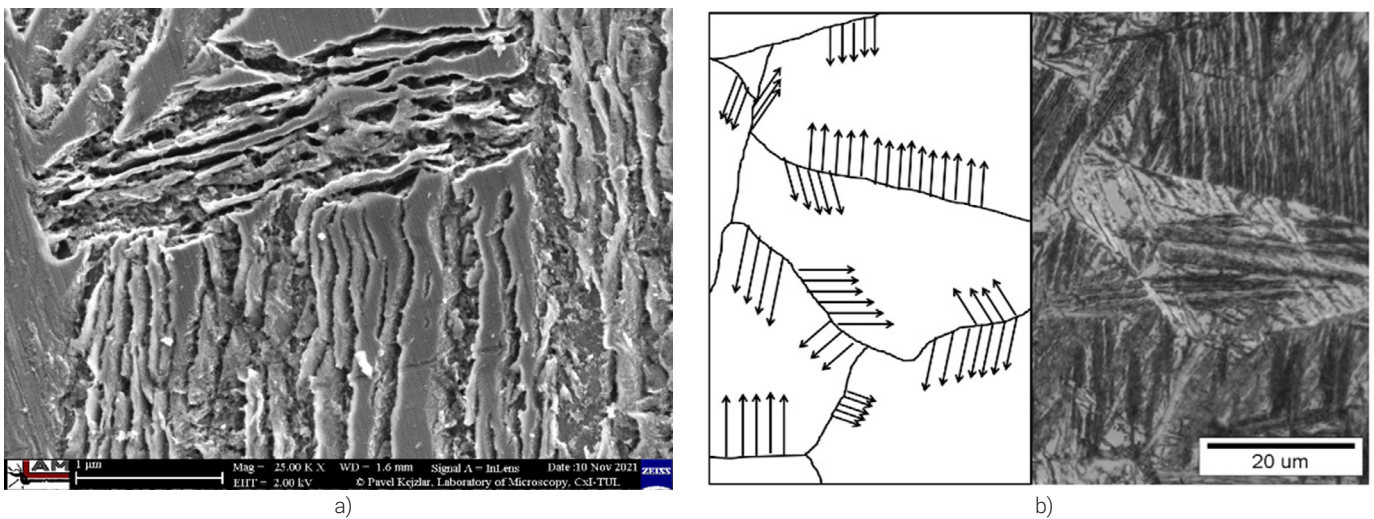

a)

b)

Fig. 5. Growth of ferrite plates on austenite grain boundaries; a) SEM visualization of the deeply etched microstructure of the mixture of ferrite plates and lamellar austenite visible in the foreground; b) growth directions of ferrite plates in primary austenite grains in a bainitized iron alloy

a)   b)   c)   d)
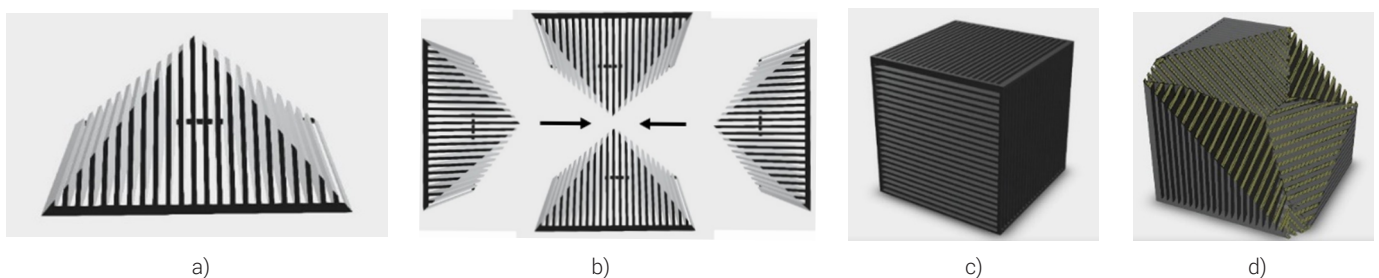
Fig. 6. Construction of the basic cell - model of a mixture of lamellar ferrite and percolating austenite: a) basic element; b) assembly of the basic cell; c) basic cell; d) cross-section through the basic cell
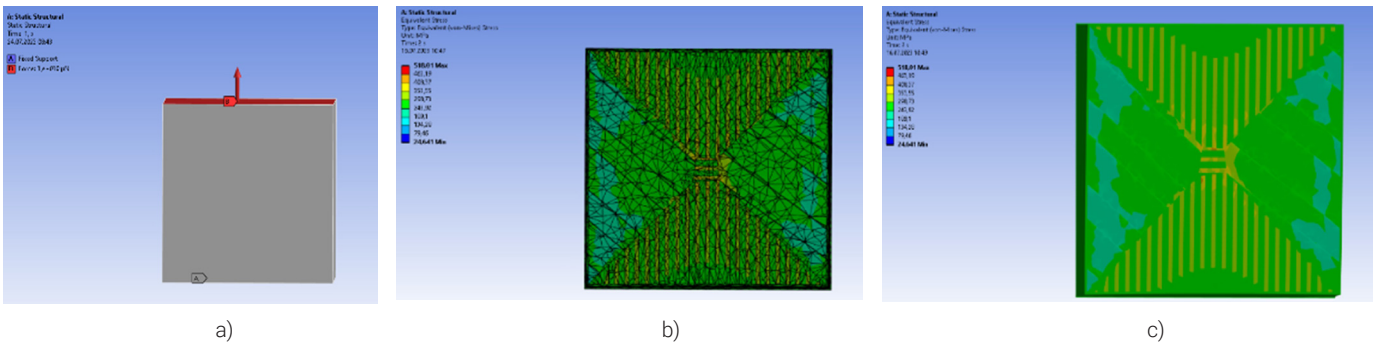
Fig. 7. FEM analysis carried out on the base cell during static stretching: a) direction of the tensile force; b) FEM mesh within the deformed base cell; c) stress distribution on the cross section of the base cell
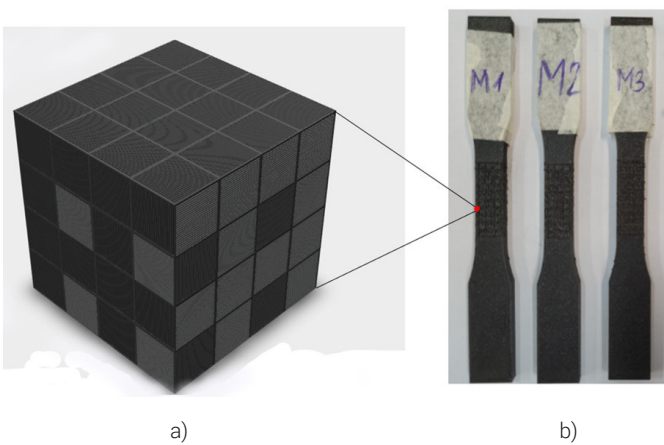

Fig. 8. A spatial model of the percolating austenite structure (empty spaces reflect the shape of the ferrite plates): a) polycrystal model; b) 3D printed tensile test specimen with a polycrystal model in the measuring part

The model assumed a specific morphology of the austenite and ferrite phases, i.e. the shape of the ferrite plates, but also their thickness, or rather the ratio of the thickness of the ferrite plates and austenite layers separating them at the level of 3:2.

The FEM analysis of the created models allows for the conclusion that, compared to a uniform structure, the basic cell system is characterized by a complex state of stress, which is divided into areas that are more and less susceptible to tensile stress. Material used in the test was SS316. The model was tensile tested by applying a force of 10,000N to the top face/plane of the cube. Fixed support was defined on the bottom face/plane of the cube. Figure 7 shows the von-Mises stress distribution, where the minimum stress value is 24.6 MPa (marked in blue) and the highest is 518 MPa (marked in red).

On the basis of the basic cell, a spatial model imitating the polycrystalline structure was built (Fig. 8a), which was then converted into the form of a strength sample by 3D printing in a polymer material (Fig. 8b). 3D printing was made on the Prusa Mini+ device using ASA filament (modified ABS, UV resistant). The static tensile test was performed on the LabTest 5.20 SP1 device at a tensile speed of 10mm/min. The results of strength tests are presented graphically in Figure 9.

Based on the conducted tests, it can be concluded that the determination of the tensile strength of a full printed structure and one that imitates the structure of a multi-phase material is possible and shows the differences in properties. Looking
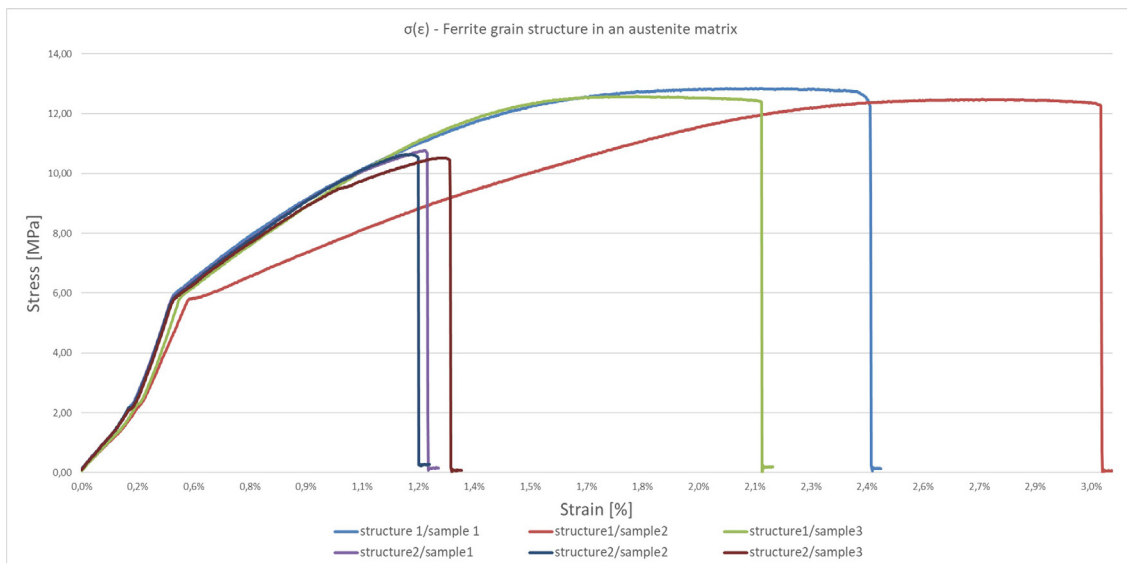

Fig. 9. Strain-stress plot of printed structure 1 and structure 2: Structure 1 is a continuous sample model; Structure 2 is a model of a sample containing a polycrystal based on a basic cell, taking into account only the mapping of the austenite phase (empty spaces reflect the shape of the ferrite plates)

closely at the tensile results of the test specimens, the strength of the percolation structure is naturally lower than that of a full 3D printed cross-section. However, it shows a clearly smaller dispersion of results than in the case of a full cross-section. This may be due to the heterogeneity of the printed structure.

Conclusions

Based on the conducted analyzes and tests, it was found that it is possible to assess the impact of percolation on the mechanical properties of multiphase materials whose structure can be determined by physical modeling. It was found that:

- In some multiphase materials, phase percolation can be identified resulting in a change in specific physicochemical properties of the material,
- Percolation can be determined by various methods appropriate for the tested material, and one of such methods can be physical modeling using modern production methods, e.g. 3D printing,
- Physical models of the microstructure of a multiphase material require a thorough understanding of the phenomena shaping its structural features, but also the geometry of the smallest elements of such

a system,

- Austenite percolates in the bainite structure and can be modeled using computer-aided techniques;
- Incremental techniques can be used to analyze the impact of the percolation phenomenon on the mechanical properties of the structure when analyzing its various variants, with the appropriate selection of print parameters and using appropriate post-print processing techniques

Determining the actual possibilities of modifying the structure using 3D printing requires an in-depth analysis and understanding of the phenomena occurring during the manufacturing process itself.

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