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Example of the methodology for producing heterostructural systems using plastic deformation

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Abstract. The study specifically examines the changes in the properties of the investigated systems as metal-to-metal composites and their constituent components. These heterostructural systems were crafted from microalloyed steel, reinforced with rheological non-uniformity due to the presence of Ti or Mg hcp phases. The influence of total deformation on the continuity of hcp layers and the degree of refinement of the matrix microstructure, i.e., microalloyed steel obtained through continuous recrystallization, was examined. The analysis delves into various strengthening mechanisms within the studied heterostructural systems, with particular emphasis on the strengthening effect resulting from the presence of low-plasticity hcp phases and their interaction with the entire system. The results of rheological studies conducted using channel compression tests and deep wire drawing (DWD) are presented. Microstructural studies have shown not only changes in the microstructure of the constituent materials but also the mutual interaction of these changes, directly affect the mechanical properties of the investigated heterogeneous materials.

Introduction

The demand for new structural materials that would meet the expectations of designers of new structural components in devices that have to operate under increasingly demanding operating conditions is a continuous challenge facing materials engineering. One of the commonly used solutions to optimize mechanical and operational properties is the use of metal-to-metal type composites [1,2]. This work proposes an innovative methodology for the fabrication of heterostructural materials characterized by an appealing combination of mechanical properties resulting from the interaction of multiple strengthening mechanisms and their synergistic effect. Heterostructured materials are defined as materials that contain heterogeneous zones that have dramatically different constitutive properties in the case of structural metallic materials or alternatively, very different physical properties in the case of functional materials [3]. According to the above definition, the materials with the following structures can be classified as typical heterostructured materials [1]: heterogeneous lamella structures, gradient structures, laminate structures, dual/multi-phase structures, harmonic (core shell) structures, multimodal structures, etc. The purpose of the present study is to outline the new strategies for structural materials development offered by new degrees of freedom and by their combination: not only playing with the microstructure or with the macroscopic shape, but allowing a new scale for materials organization, the “architecture”, and controlling a new degree of freedom, the “spatial heterogeneity,, [4]. The presented research discusses the rheological properties and the results of microstructural analysis of multilayer systems produced through the channel test and deep wire drawing (DWD). The analysis of results obtained in channel die compression tests allowed for the identification of conditions for conducting deep drawing processes of wires, to take into account all interactions arising from both the inhomogeneity of strengthening processes and microstructural changes.

Experimental

The presented studies aimed to produce mechanically and structurally inhomogeneous materials and investigate the microstructural changes and mechanical response of such multilayered systems. Two deformation processes were employed for the multilayer systems composed of microalloyed steel and titanium (St/Ti) or magnesium (St/Mg): channel test and deep wire drawing (DWD). Both applied deformation methods allowed the accumulation of significant plastic deformation energy in the deformed materials, resulting in non-uniform strengthening through an increased density of geometrically necessary dislocations (GND) revealed using electron backscatter diffraction *(EBSD)* based on kernel average misorientation *(KAM)*. Experimental research included tests of packet compression in the channel test and processes of wire drawing with very large total deformations. In Fig. 1, the experimental setup for channel tests is shown, with samples constructed with a packet arrangement of layers and an illustrative diagram of the drawing process of inhomogeneous wires with the concept of creating the structure of the matrix material (microalloyed steel, bcc) reinforced with macro and meso particles of a less ductile phase (Ti, Mg, hcp). Published earlier studies [5, 6] have shown that the microalloyed steel with the addition of Nb, used in the presented research, is a suitable material for very large deformations and for the generation of Severe Plastic Deformation (SPD) effects in the examined heterostructural wires. In the case of channel compression tests, packages consisting of two layers of microalloyed steel were compressed, with a layer of Ti or Mg between them (Fig. 1b). All samples had a length of 15mm and a width of 10mm. Two thicknesses of the steel layer were used, namely 4.0 mm and 2.0 mm, along with spacers of Ti or Mg with a thickness of 1 mm. These prepared packages underwent the compression process with two total strain magnitudes of 0.25 and 0.5. For the DWD processes, the procedure involved wire drawing a system with a circular cross-section, constructed by placing a rod of Ti or Mg with a diameter of 3 mm in a hollowed microalloyed steel cylinder with a diameter of 6.5 mm (Fig. 1c, d). The multi-stage wire drawing process was carried out with a unit reduction of cross-sectional area equal to 15%, until the maximum possible reduction in the diameter of the investigated multilayer system was achieved. Additionally, wire drawing processes were carried out in the same multi-pass scheme using a “homogeneous” microalloyed steel rod with an initial diameter of 6.5mm. In this case, the wire drawing process was executed until achieving a diameter reduction to 2.5mm. Some wires were drawn to a diameter of 0.75mm. Intermediate annealing was not applied between drawing operations. The resulting research materials underwent microstructural analysis and mechanical property testing i.e. tensile tests and hardness measurements.

The research materials used had the following characteristics:

- *Microalloyed steel*: bcc, Shear Modulus: G = 78 GPa, Chemical composition, (wt%): 0.07C/0.29Si/1.36Mn/0.067Nb/0.03Ti/0.16Cu/0.009N//Fe – bal. Initial grain size: 22 μm.
- *Ti, grade 2*: hcp, Shear Modulus: G = 43 GPa, Chemical composition, (wt%): 0.01Fe/0.03H/ 0.04O/0.01N/Ti-bal. Initial grain size: 28 μm.
- *Mg, AZ31*: hcp, Shear Modulus: G = 17 GPa, Chemical composition, (wt%): 3.27Al/0.98Zn/0.28Mn/0.0027Fe/0.00078Ni/0.11Si/Mg- bal. Initial grain size: 20-25 μm.

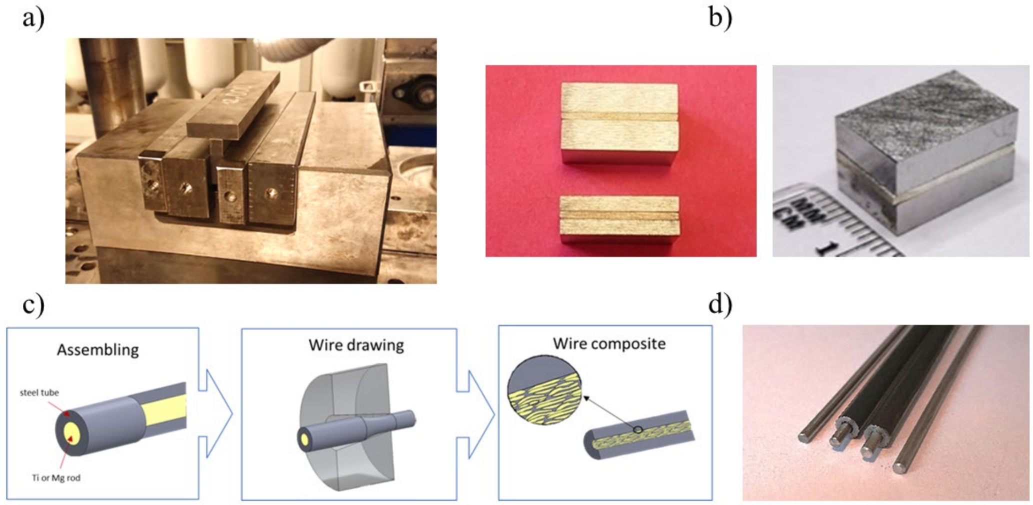

Fig. 1. Research methods employed in the production of the studied multilayer and heterostructural systems. Test stand for channel testing (a); constructed multilayer samples for channel compression (b). Diagram of heterostructural material production using the deep wire drawing (DWD) process (c). Inhomogeneous wire samples for DWD tests (d).

The impact of the scale of microstructure elements participating in the strengthening of the examined systems was considered, taking into account the following factors: i) the degree of reinforcement achieved by developing the matrix microstructure (grain refinement and perlite colonies) and the second phase (particles of fragmented Ti or Mg layers produced by wire drawing and deep wire drawing, ii) the introduction of new deformation mechanisms due to the presence of incoherent second phase particles on the micro-meso scale, iii) changes in competitive processes, such as the loss of coherence and healing processes, cracking events, or recovery processes, accompanying the very strong deformation of the studied heterostructural multilayer systems.

Results and discussion

The first stage of the research involved testing by the channel compression tests of multilayer systems. The aim of these tests was to obtain information necessary for the proper design of the second stage of experimental research, namely the deep drawing processes of heterostructured wires. Example results of computer simulations and microstructure analysis, using EBSD technique, of the multilayered sample (steel/Ti/steel) are presented in Fig. 2. Suitable rheological and microstructural models have been proposed, the application of which can rationalize the behavior of the studied materials by considering different dimensional scales and allowing for interactions between incoherent phases of materials used in the studied metal-to-metal composites. As seen in Fig. 2a, the deformation of the compressed sample and the non-uniform distribution of effective strain translate into the degree of grain deformation in the layers of steel, i.e., stronger effects are visible in the upper layer, which was influenced by the moving hammer. This observation is significant from the planning-optimization perspective of wire drawing processes for heterostructured wire systems, where the forces in the deformation zone originate from the cone of the drawing die, causing typical non-uniformity in plastic deformation zone with all the consequences in microstructural changes. In Fig. 2b, the intensity of strain energy accumulation is shown, represented by the degree of grain deformation and distributions of geometrically necessary dislocation densities as an image of KAM (Kernel Average Misorientation). For the applied thickness of the steel layers (4.0 mm) and the deformation magnitude of the studied system (0.25), a noticeably stronger work hardening is evident in the Ti layer. It can be presumed that the direct cause of this is the lower plasticity of Ti, due to its hcp structure.

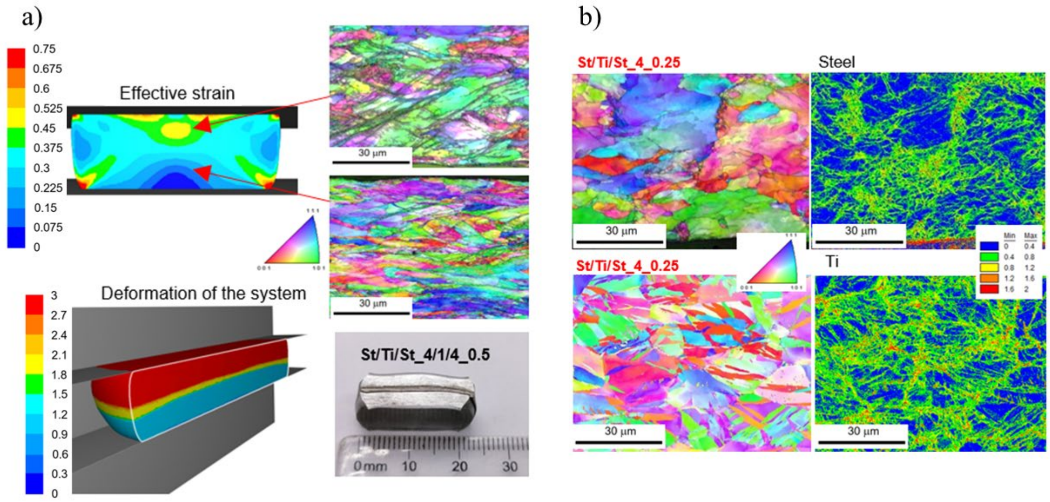

Fig. 2. Example results of computer simulations and microstructure analysis of the multilayered sample (steel/Ti/steel), using EBSD technique (a). Representation of strain hardening in adjacent layers of steel and Ti, showing, for a given package (steel layer thickness 4.0 mm) and strain magnitude (0.25), a faster increase in the density of geometrically necessary dislocations, represented by KAM, in the titanium layer (b).

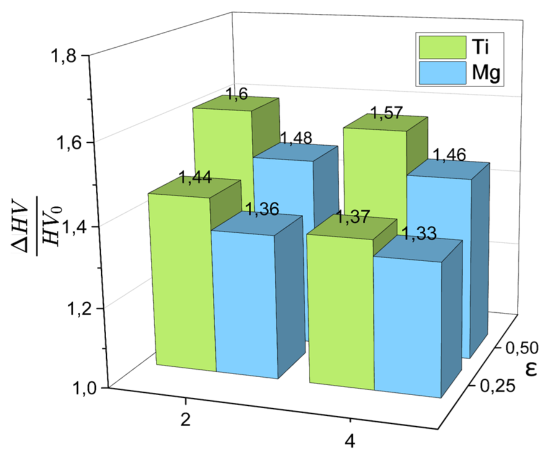

Fig. 3. Relative hardness increases of Ti and Mg as a function of the thickness of the steel layers and the total deformation of the packages produced in the channel compression tests.

Understanding the characteristics of the strengthening processes in Ti or Mg layers is crucial for designing the heterostructural systems targeted in the current research. Increased work hardening (relative hardness increase) was significantly observed in the Ti layers compared to Mg, especially when higher deformation was applied (Fig. 3). In-depth studies in this area have also been presented in previous publications by the authors [7,8]. From these observations, a direct conclusion can be drawn that Ti, due to more effective accommodation of deformation effects, may be a more effective layer in creating dispersed particles resulting from the loss of coherence in a highly deformed heterostructural system (see Fig. 1c).

In the second stage of the research, heterostructural materials produced through deep multistage wire drawing were investigated. In this process, deformation effects corresponding to reductions in diameters to 4.0 mm and 2.5 mm were accumulated. Wires with structurally non-uniform characteristics were examined, consisting of a layer of steel and a titanium core (see Fig. 1c,d). Mechanical property studies and microstructural analysis were conducted, allowing for the identification of the fundamental causes behind the observed mechanical responses of the investigated materials. In Table 1, the mechanical properties of heterostructural wire systems with diameters of 2.5 mm, featuring cores made of Ti and Mg, are presented. For comparison, the results of tensile tests on a homogeneous wire made of microalloyed steel with the same deformation history are also included. Significant differences in the strength and plastic properties of the investigated wires can be observed. The ability to formulate more general conclusions from the research on the mechanical properties of produced heterostructural wires requires further investigations with an increased degree of deformation. This is necessary to achieve the expected microstructural effects in the form of Ti core fragmentation, which is the overarching goal of the presented studies.

Table 1. Mechanical properties of the investigated "homogeneous" steel wires and heterostructural wire systems of Steel/Ti and Steel/Mg.

Type of system, ϕ 2.5 mm	**YS [MPa]**	**TS [MPa]**	**EL [%]**	**RA [%]**
Steel	1153	1236	1.90	37.00
St/Ti	1007	1227	5.51	21.56
St/Mg	800	818	3.85	5.66

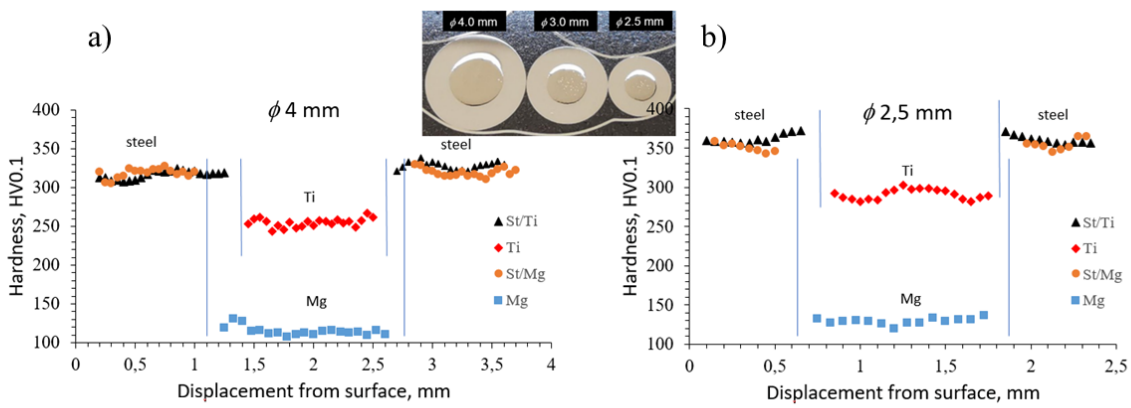

Fig. 4. Hardness distributions in the layers of steel and Ti or Mg on the cross-section of heterostructural wires after reducing their diameter to 4.0 mm (a) and 2.5 mm (b).

Undoubtedly, a crucial aspect of the discussion regarding the results obtained at this stage of the research is the assessment of the non-uniformity of strain strengthening in the examined heterostructural systems. The direct representation of the strain strengthening characteristic is hardness distributions on cross-sections of produced wires. In Fig. 4, the hardness measurement results for steel/Ti systems deformed by reducing diameters to 4.0 and 2.5 mm are presented. The presented hardness distributions in the layers of steel, Mg, and Ti confirm earlier observations from the first stage of the study, specifically the channel die compression tests, where an increase in hardness was noted in the steel layers near the contact with the Ti layer, especially for higher effective strains. No such effect was observed in the case of the Mg system.

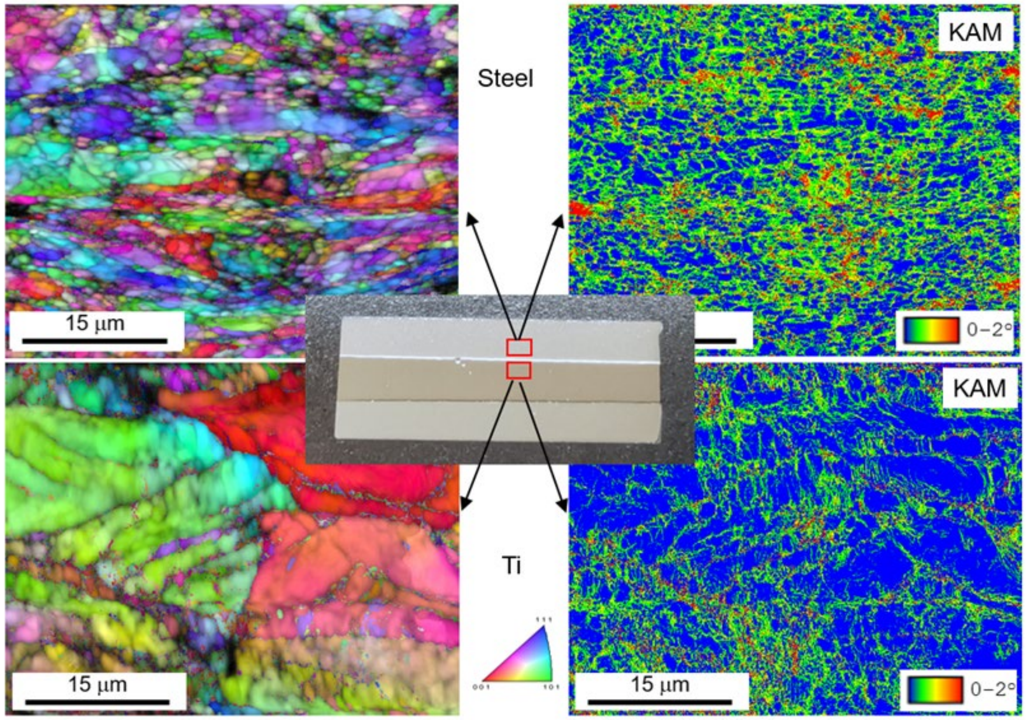

Fig. 5. Results of microstructural investigations using EBSD and KAM analysis in the contact zone of the steel and Ti layers on the longitudinal cross-section of the heterostructural wire after reducing the diameter to 4.0 mm.

The results of microstructural analysis presented in Fig. 5 show that even after reducing the diameter to 4.0 mm, a distinct separation of work hardening is observed between adjacent materials, characterized by different ductility. The ability to accommodate deformation is represented in this case by KAM maps showing the geometrically necessary dislocation densities. In the case of the steel layer, the effects of initiated continuous recrystallization can be observed, resulting in clear colonies of a fine-grained structure. It is expected that this *in situ* recrystallization process, as a result of strong accumulation of plastic deformation energy, will progress with increasing levels of deformation in subsequent passes. In the presented microstructures, it is evident that in adjacent regions of the steel and Ti system, the former exhibits a higher degree of work hardening. This means that in the case of wire drawing heterogeneous material systems, such as the investigated steel/Ti samples, after reaching a certain degree of hardening, the Ti core begins to function as a specific tool for the more ductile steel layer. Confirmation of this is also found in observations of the relative change in the thickness of the steel layer to the diameter of the Ti core. It was noticed that the reduction in the diameter of the entire heterogeneous steel/Ti wire, with the increase in total reduction, occurred to an increasing extent at the expense of reducing the thickness of the steel layer. From this, the conclusion can be drawn that, in order to ensure the possibility of further deformation through the drawing process of the wire until achieving a core thinning of Ti to the point where its fragmentation begins, leading to the formation of fine particles, it will be necessary to introduce interoperative annealing.

In Fig. 6, microstructure images of etched longitudinal sections of steel/Ti wires deformed by reducing the diameter to 2.5 mm are presented. Clear deformed grains of both steel and Ti can be observed. From these observations, it can be inferred that the steel/Ti wires constitute a suitable research material for further studies on intense deformation of heterostructural systems, until the expected strengthening effect by Ti particles formed from its fragmentation due to the loss of coherence after utilizing the entire plasticity is achieved. The subject of further research will also be the condition of the connection between the layers of steel and Ti at various stages of total deformation in consecutive sequences, down to reducing the diameter below 0.75 mm. Previous studies [6] have shown that it is possible to produce in homogeneous wires from the investigated microalloyed steel, nanostructures with an average grain size of about 250 nm as a result of the in-situ recrystallization process during Severe Plastic Deformation (SPD).

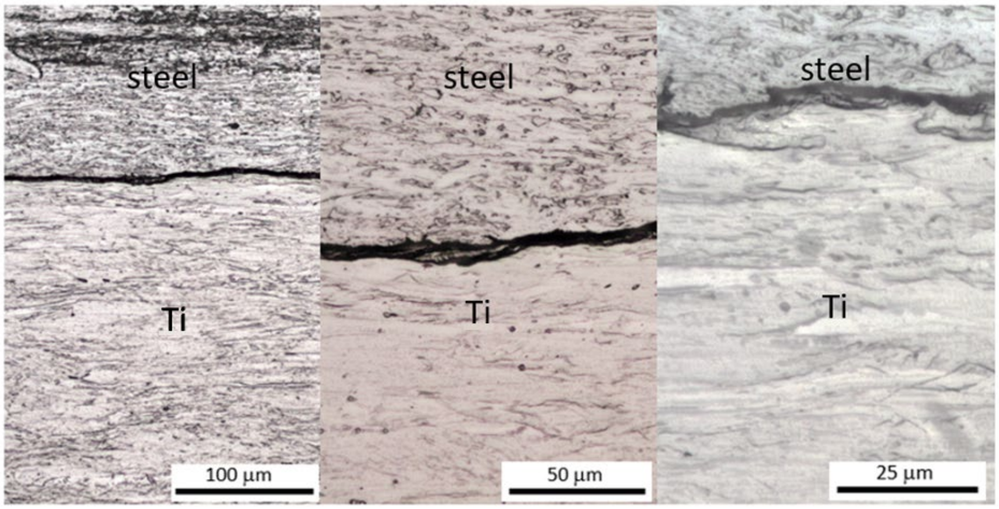

Fig. 6. Microstructures at the interface of the steel layer and Ti of the heterostructural wire after reducing the diameter to 2.5 mm, at various magnification scales. Cross-section.

Summary

The conducted plastometric and microstructural studies have shown that there is a significant variation in work hardening effects not only in adjacent layers of microalloyed steel and Ti or Mg, but also as an effect of the magnitude of the total applied strain and the positioning of these layers in a multilayer stack. The obtained results were used to plan and carry out Deep Wire Drawing (DWD) processes for the production of heterostructural systems. The experimental studies were supported by computer simulations, allowing for the assessment of mechanical states accompanying the investigated deformation processes, such as channel die compression tests and wire drawing processes. The effects of the mutual interaction of the constituent layers of the studied heterogeneous systems have been presented.

The main cognitive effects of this work were achieved through exploring the possibilities of new processes for manufacturing multilayer systems based on microalloyed steels, which can ultimately be reinforced with particles of hcp phases with low ductility and varying degrees of dispersion. The research results will be utilized to design subsequent stages of the study, aiming to produce new multilayer drawn products with attractive combinations of strength and ductility.

Acknowledgment

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