Material Forming - ESAFORM 2024 Materials Research Forum LLC
Materials Research Proceedings 41 (2024) 733-741 https://doi.org/10.21741/9781644903131-81

Heterostructures produced through severe plastic deformation of multilayered systems: Steel-Ti and steel-Mg

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Keywords: Heterostructured Materials, Multilayer Composite, Multi-Stage Wire Drawing

Abstract. The presented research encompasses the processes of manufacturing incoherent metal-to-metal composite materials based on a multi-layered system. The primary aim of the present research is to achieve heterostructured materials with small "hard" phase particles within the "soft" phase matrix. Particles of the "hard" material can be obtained as a result of the loss of their continuity, for example, due to severe plastic deformation effects, such as accumulative roll bonding or multi-stage deep wire drawing processes. The presented research results constitute the first stage of implementing the aforementioned research plan. Basic relationships between the parameters of the deformation process and microstructural changes, as well as rheological properties of the investigated components of multilayer systems, i.e., microalloyed steel, Ti, and Mg, were determined. For this purpose, channel die compression tests and multi-stage deep wire drawing (DWD) experiments were conducted. The microstructural examinations and mechanical properties of the produced materials have indicated directions for further research.

Introduction

Continuous development and the increasing demand for new structural materials necessitate the design of materials that exhibit both high strength and good ductility. Since the mechanical properties of metals and alloys are determined by their deformation and strengthening mechanisms, it is essential to base the design of new structural materials on modifying and improving these mechanisms. Therefore, the proposal to combine materials with different workabilities and structurally incoherent multilayered components, further varying in microstructure component morphology, enables the attainment of an attractive combination of mechanical properties [1,2].

The main focus of the present study is to generally combine several mechanical and microstructural aspects to produce new structural multilayered materials, based on inhomogeneous deformation of incoherent metal-to-metal composites, i.e., microalloyed steel (bcc) and Ti or Mg (hcp), with specific properties. The presented results are part of the preliminary research for an ongoing research project on multilayer systems, with a specific focus on the phenomena occurring at the interface of the bcc and hcp phases. The influence of such a diverse microstructure is being investigated in terms of the following factors:

i) The level of strengthening achieved by refining the structures of the matrix (grains, pearlite clusters) and the second phase (particles of fragmented Ti or Mg layers) through compression tests and deep wire drawing (DWD).

ii) The introduction of new deformation mechanisms due to the presence of incoherent second-phase particles at the micro-meso scale.

iii) Changes in competitive processes such as fracture or recovery events, which are related to the structural refinement of multilayered, inhomogeneous metal-to-metal composites.

The procedure involved preliminary research of the channel tests and multi-stage wire drawing of materials composed of layers (Fig. 1) with different workabilities at room temperature, including microalloyed steel, Ti alloys, and Mg alloys. Previous studies [3] demonstrated that microalloyed steel with a high Nb content is characterized by both high strength and good ductility[4-7]. More ductile matrix material, i.e., microalloyed steel, due to complex strengthening mechanisms (*Considere* criterion) during severe plastic deformation and due to accumulated deformation energy, dislocation density, undergoes significant microstructure refinement through *in situ* recrystallization.

The presented results are part of a project in which various combinations of steel/Ti alloy/steel and steel/Mg alloy/steel systems underwent deformation in channel tests [8]. Additionally, the drawing process of the tube/rod system was carried out. The discussion of the results is based on microhardness measurements, tensile tests, optical microscopy, and analysis using the Electron Backscatter Diffraction (EBSD) technique to assess microstructural changes. Plastometric tests and microstructural investigations are complemented by representations of mechanical states in the examined multi-layered systems.

Experimental

In the present studies, two deformation processes were employed for multilayer systems composed of microalloyed steel and Ti (St/Ti) or Mg (St/Mg): channel test and deep wire drawing (DWD). Both methods of deformation application allow the accumulation of strong plastic deformation energy in the deformed materials, represented by the induced strengthening through an increased density of geometrically necessary dislocations (GND).

In Fig. 1, schematics, and real images of the research setups, as well as the concepts of the applied deformation processes, are presented. 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. 1a). All samples had a length of 15mm and a width of 10mm. Two thicknesses of the steel layer were used, namely 4 mm and 2 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 (Tab.1). 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. 1b). 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 1.96mm (Tab. 2). 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, including hardness and tensile tests.

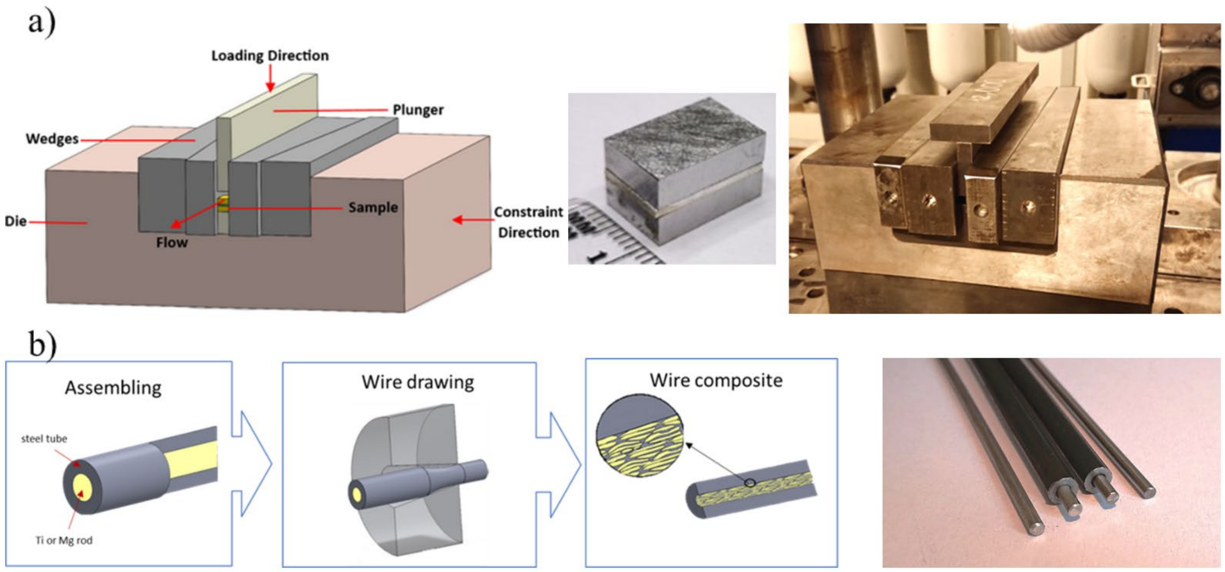

Fig. 1. Schematic and real representations of the channel test (a) and the drawing process of the multilayered (tube/rod) composite (b).

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/0.0005Cu/0.11Si/Mg-bal. Initial grain size: 20-25 µm.

Table 1. Dimensions of samples subjected to channel compression tests and their corresponding effective strains. The sample names contain information about the thickness of the steel layers.

Package type	Initial package height, H_0 [mm]	Compressed package height, H_f [mm]	Effective strain $\varepsilon=\ln(H_0/H_f)$
Idealized dimensions	5,00	3,89	0,25
	9,00	5,46	0,50
St/Ti_2	5,01	3,92	0,245
St/Ti_2	5,02	3,10	0,482
St/Ti_4	8,98	6,90	0,263
St/Ti_4	8,95	5,38	0,509
St/Mg_2	5,08	3,85	0,277
St/Mg_2	5,10	3,18	0,472
St/Mg_4	9,09	6,91	0,274
St/Mg_4	9,06	5,58	0,485

Table 2. Diameters and magnitudes of deformations obtained in individual passes of wire drawing, as well as the magnitudes of total accumulated effective strains.

No. of pass	Diameter of the die, D_f [mm]	Reduction of area RA/pass, [%]	Effective strain/pass $\varepsilon=2\ln(D_o/D_f)$	Accumulated effective strain
0	6,5	-	-	-
1	6,2	4,6	0,09	0,09
2	5,9	4,8	0,10	0,19
3	5,6	5,1	0,10	0,30
4	5,4	3,6	0,07	0,37
5	5,2	3,7	0,08	0,45
6	5,0	3,8	0,08	0,52
7	4,8	4,0	0,08	0,61
8	4,6	4,2	0,09	0,69
9	4,3	6,5	0,13	0,83
10	4,0	7,0	0,14	0,97
11	3,6	10,0	0,21	1,18
12	3,2	11,1	0,24	1,42
13	2,8	12,5	0,27	1,68
14	2,5	10,7	0,23	1,91
15	2,18	12,8	0,27	2,18
16	1,96	10,1	0,21	2,40

Results and Discussion

The objective of this research stage was to evaluate the intensity and inhomogeneity of work hardening, as well as microstructure evolution, in the examined multilayered materials (steel, Mg, Ti) under various conditions of deformation, including variations in layer thickness and layer placement location. It is expected that the results of the assessment of the behavior of the tested materials in multilayer systems in the macro-scale will allow for a more precise selection of conditions and the construction of metal-to-metal composites in further stages of the project, i.e., study at the meso and micro scales.

Channel compression test. The first stage of the research involved conducting channel testing to define the basic relationships between the fundamental characteristics of the cooperating layers in the examined layer packages and microstructural changes as well as mechanical properties. The packages subjected to the study varied in the thickness of the cooperating layers of steel, Ti or Mg, as well as in the magnitudes of the total deformation imposed during the compression process.

The diagram of the testing system, used in the studies of channel compression, is presented in Fig. 1. In Fig. 2, the relative hardness changes are presented, both in the layers of steel and in Ti or Mg, as representations of strain hardening in specimens subjected to channel die compression tests. Hardness measurements were conducted with loads of 0.1 kG. The measurement was performed for compressed layers in packages.

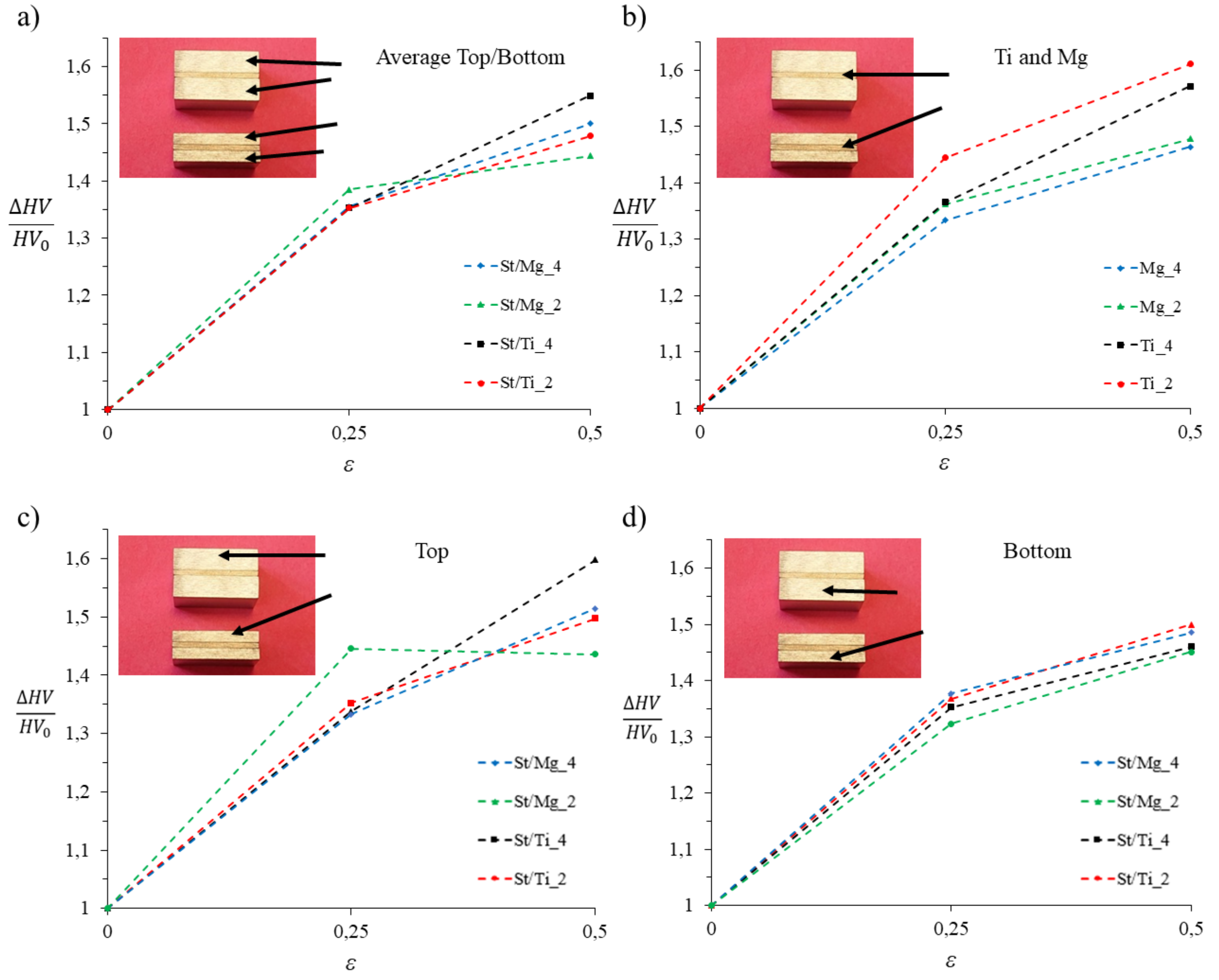

Fig. 2. Changes in the relative hardness (an average value in the layers) of steel (a), Ti and Mg layers (b), as a function of the applied effective strain. Effect of the steel layer's location in the pack (top, bottom) (c) and (d), during channel compression test. The thickness of steel layers are indicated in the legend as: St/Mg_2, St/Mg_4 or St/Ti_2, St/Ti_4 (2mm or 4mm respectively).

From the presented results comparison, it can be observed that the location of the steel layers did not show a considerable influence on their work hardening (Fig. 2c,d). Nevertheless, for larger deformations, the upper layer tends to exhibit greater strengthening (Fig. 2c). Increased work hardening (relative hardness increase) was significantly observed in the Ti layers compared to Mg, especially when higher deformation was applied.

For small deformations and thicker layers of steel, stronger effects of work hardening (GND density) were observed in the Ti layer than in the steel (Fig. 3a). The analysis of the KAM (Kernel Average Misorientation) showed also a slightly greater increase in GND density in the upper steel layers near the Ti contact, even in the case of small thicknesses of steel layers (Fig. 3b). This shows that one can expect greater hardening effect in layers located farther from the axis of drawn inhomogeneous wires.

The results presented in Fig. 2 and Fig. 3 allow for the selection of appropriate thicknesses for individual layers in an inhomogeneous steel/Mg or steel/Ti drawing system, in order to optimize the plastic flow of the entire system during wire drawing processes in the next stage of research.

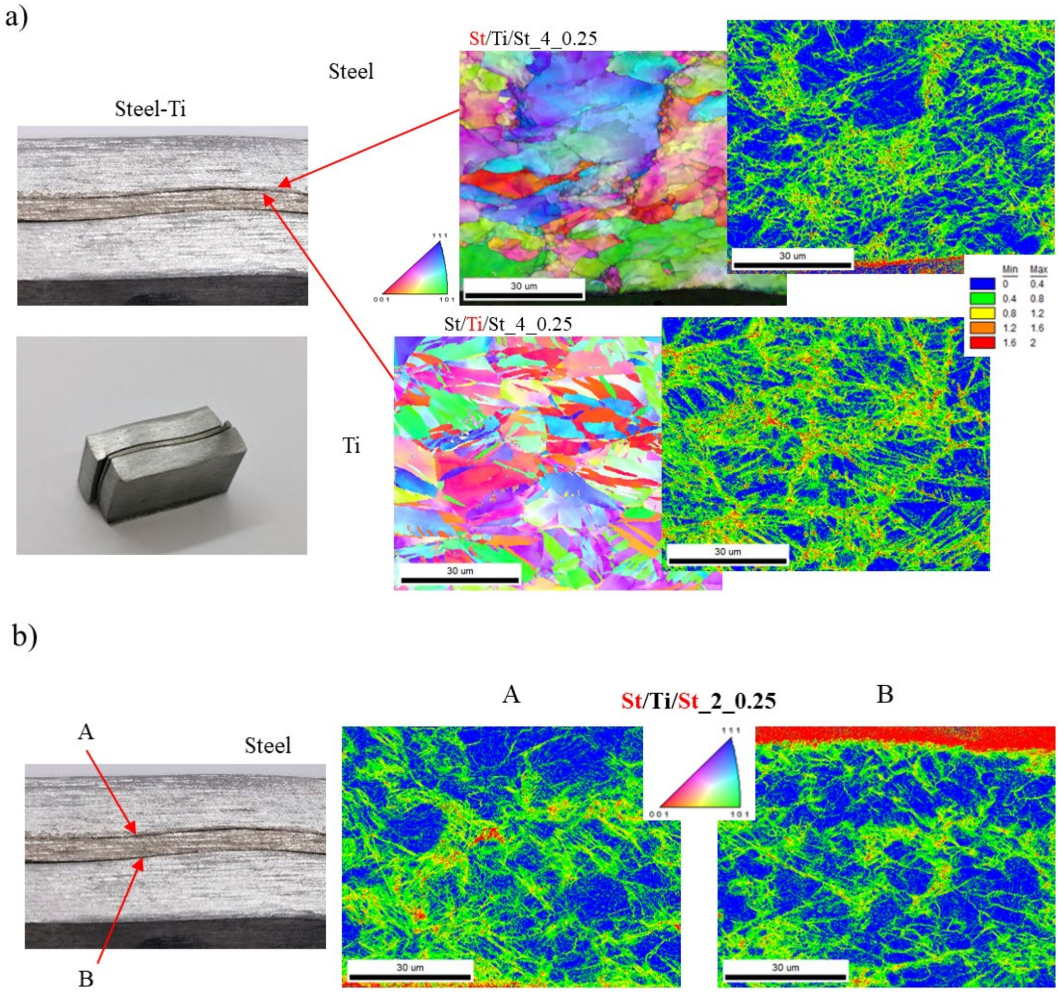

Fig. 3. Example of results from microstructural studies using EBSD analysis. KAM analysis revealed different increases in the density of GND (geometrically necessary dislocations) in the layers of steel and Ti as representations of differences in work hardening states –(a) and the variation in the degree of work hardening in the layers of steel due to their position in the stack – (b). St/Ti/St_4_0.25 denotes a system composed of steel and Ti with a steel layer thickness of 4mm deformed with an effective strain of 0.25.

Deep Wire Drawing (DWD). The second part of the study focused on the processes of wire drawing heterogeneous systems, as presented in Fig. 1b. Inside the microalloyed steel wire rods, smaller rods with a diameter of 3mm were placed. The system with rods made of Ti or Mg was studied. Simultaneously, deep wire drawing processes of homogeneous wires from microalloyed steel were carried out. In this case, the results confirmed earlier research findings [9], which demonstrated that by using the applied DWD process, effects characteristic of Severe Plastic

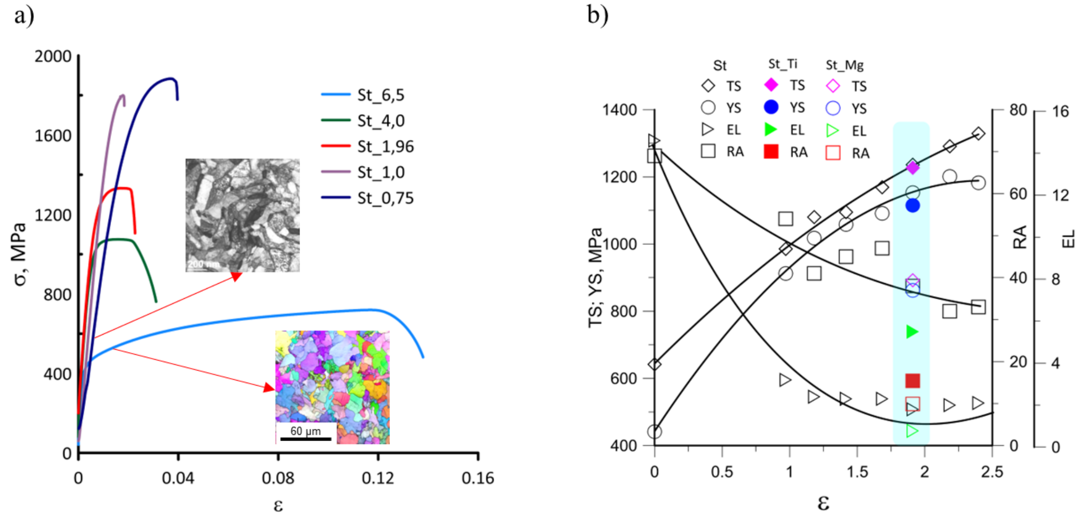

Fig. 4. Characteristics of the base material in a multilayer system – microalloyed steel, in terms of work hardening and the resulting changes in mechanical properties of wires after multi-stage drawing. Samples taken from "homogeneous" wires drawn to specified diameters, in range of 6.5mm-0.75mm – a) and comparison of the mechanical properties of "homogeneous" (St) samples with systems containing Ti (St/Ti) or Mg (St/Mg), as a function of the total effective strain after multistage wire drawing- b).

Deformation (SPD) could be achieved. In Fig. 4a, the changes in mechanical properties and microstructure of the "homogeneous" wires after various diameter reductions obtained in multi-stage wire drawing without interoperative annealing are presented. As a result, an average grain size of approximately 300nm was achieved at a diameter of 0.75 mm. The presented results confirm the appropriateness of selecting microalloyed steel as the base material in heterogeneous systems, i.e., a material that will exhibit very good plasticity and susceptibility to in situ recrystallization.

In Fig. 4b, the changes in mechanical properties corresponding to different total accumulated effective strain in drawn homogeneous wire rods and those with Ti or Mg rods for a strain of 1.96 (highlighted in the chart by a blue bar) are shown. The obtained results indicate significant differences in plastic properties (RA, EL) between the studied materials. The observed changes in mechanical properties significantly differ from those obtained for steel wires, specifically in terms of strength properties for the St/Mg system and plastic properties for the St/Ti system. Nevertheless, further research is necessary to draw general conclusions. Currently presented results of the study on rods subjected to the DWD process, both homogeneous steel and those reinforced with Ti or Mg rods, indicate significantly different microstructural changes in the tested materials (Fig. 5). It was demonstrated that in the St/Ti system (Fig. 5a, b), the development of the microstructure, due to the position of material layers in the cross-sectional view, shows similarity to the results obtained in channel compression tests. An important observation is also the limitation of the total possible deformation in the St/Mg system. It was noticed that at a package diameter of 2.5 mm, and even larger, typical effects of coherence loss occurred inside Mg rods during drawing processes (Fig. 5c). Hence, the conclusion is drawn that Mg is less suitable for building structurally heterogeneous systems where strong deformation accumulation is expected to produce fine reinforcing particles in the matrix of ultrafine-grained micro-alloyed steel.

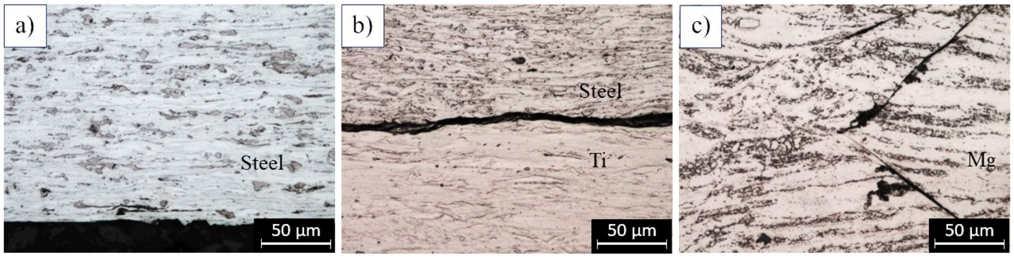

Fig. 5. Microstructures after the DWD, deformed to a diameter of 2.5mm. Image of the base from microalloyed steel in the near-surface layer (a) and in the contact zone with the Ti rod (b). Typical image of material coherence loss subjected to wire drawing (St/Mg).

Summary

The current work has revealed the complexity of the rheological properties and microstructure development in the investigated multilayer systems. It has been demonstrated that the variation in the thickness of the matrix layers, i.e., steel in the steel/Ti/steel and steel/Mg/steel multilayer systems, has an influence on the plastic flow of the entire multilayer system. A direct consequence of this situation is the inhomogeneity of work hardening in individual layers and the localization of microstructure deformation. This is expected to result in the distribution of dislocation densities (GND) and fracture micro-mechanisms, leading to the disintegration of the Ti and Mg layers into incoherent particles with varying dispersion.

It has been shown that the experimental results of DWD correlate well with effects observed in the channel tests regarding the mechanical response of the investigated multilayer systems. The results of the research will be used to design the next stages of the study, i.e., the composition of pipe-rod and pipe-pipe-rod systems, in order to produce new multilayer drawn products with attractive combinations of strength and ductility.

Acknowledgment

Research financially supported by National Science Centre, Poland, Grant No 2022/45/B/ST8/01383

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