Characterization of Cu-Al-Ni melt-spun ribbons using a focussed ion beam (FIB)

Karakterizacija hitrostrjenih trakov zlitine Cu-Al-Ni z uporabo fokusiranega ionskega curka

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Abstract: This work investigates the possibilities for applying a focussed ion beam (FIB) for the metallographic preparation and characterization of Cu-Al-Ni melt-spun ribbons. Two alloys were selected for this reason: CuAl13Ni4 and CuAl15Ni4. The microstructure of the first alloy was fully martensitic and the microstructure of the second consisted of two phases: martensite and γ_2. It was discovered that with FIB-etching the microstructures of both alloys can be clearly revealed on polished cross-sections of the melt-spun ribbons, as well as on their wheel-side and air-side surfaces. However, better results were obtained when the etched surface was smoother, and finer details were visible when using smaller ion currents. In addition, a study was made into the influence of platinum deposition on the quality of 3D-cross sections. It was found that Pt-deposition is necessary when the edge of the trench should be straight and sharp, and the surface of the 3D cross-section smooth. However, in this case, the microstructure of the ribbons free surface cannot be seen.

Izvleček: V tem delu smo raziskali možnosti uporabe fokusiranega ionskega curka (FIB) pri metalografski pripravi in karakterizaciji hitro strjenih zlitin Cu-Al-Ni. Izbrali smo dve zlitini: CuAl13Ni4 in CuAl15Ni4. Mikrostruktura prve zlitine je bila v celoti martenzitna, medtem ko je bila mikrostruktura druge zlitine sestavljena iz dveh faz: martenzita in γ_2. Ugotovljeno je bilo, da lahko pri jedkanju z ionskim curkom odkrijemo mikrostrukturo tako na poliranih prečnih prerezih trakov, kot tudi na obeh prostih površinah hitrostrjenih trakov. Mikrostruktura se je boljše odkrila, če je bila raziskana površina bolj gladka, medtem ko smo lahko razločili drobnejše mikrostrukturne

sestavine pri uporabi manjših ionskih tokov. Poleg tega smo študirali tudi vpliv nanosa platine na kakovost 3D-prečnih prerezov. Ugotovili smo, da je nanos platine smiselno uporabiti, če želimo zelo ravne in ostre robove reza ter gladko površino prečnega reza, vendar pa v tem primeru ne moremo videti mikrostrukture proste površine.

Key words: focussed ion beam (FIB), metallography, melt-spinning, shape memory alloy, Cu-Al-Ni
Ključne besede: fokusiran ionski curek (FIB), metalografija, litje na vrteče kolo (melt-spinning), zlitina z oblikovnih spominom, Cu-Al-Ni

INTRODUCTION

The traditional metallographic preparation of samples for both light microscopy and scanning electron microscopy (SEM) most frequently consists of hot or cold mounting of a sample, grinding, polishing and, finally, etching. Mounting in resin is unavoidable for small samples such as melt-spun ribbons. Therefore, as a rule, only one surface (or cross-section) of each small sample can be prepared and investigated.

The metallographic preparation of Cu-Al-Ni shape memory alloy (SMA) melt-spun ribbons is usually afflicted with problems from the very start. The as-cast microstructure may already contain the martensitic phase, its fraction being dependent on the exact chemical composition (LOJEN, 2005). Therefore, the alloy might exhibit the shape memory effect when the temperature during hot mounting varies within the range of the alloy's transformation temperatures. As a result, a sample may change its shape or even break-apart as it is held in position by clamps. Cracks in the hot-mounted samples are often observed, especially in Cu-Al-Ni alloys with relatively high Al-content. In the case of cold-mounting, during polymerization, the temperature can also reach the level of the alloy's transformation range. Additionally, gaps between the sample and the resin occur quite frequently, from which the etchant is extremely hard to remove. In our experience good universal etchants which reveal the phase composition, as well as the martensitic pattern, contain quite aggressive and volatile ingredients such as $(NH_4)_2S_2O_8$ and HCl. Consequently, it is very difficult to find the optimal dilution and to obtain reproducible results even within a time period of a few hours. Samples must be examined immediately after etching, otherwise the remnant etchant from the almost always present gaps could damage the sample before examination. All these facts stimulate the search for alternative preparation techniques. One of the most promising methods seems to be preparation using the focussed ion beam (FIB).

The FIB systems, in principle, work in the same way as the electron beam systems. Both consist of an emission source (electron or ion source), lens column, workstage, vacuum unit, and control systems. Ion optics is very much like electron optics. Both, electrons and ions are charged

particles and can be focussed into a fine beam using electromagnetic fields. The only difference is that ions can have different masses and charges (Selinger, 1979; Orloff, 2003; Gianuzzi, 2005; Zupanič, 2006).

During the scattering process, ions lose their energy and stop in the surface region of the sample. Elastic scattering changes the direction of the incident ion without a loss of energy. On the other hand, inelastic scattering causes loss of energy in two ways. One is nuclear loss, i.e. an incident ion collides with an atomic nucleus causing the target atom either to dislocate its position (a recoil atom), or to escape from the solid surface. The phenomenon of knocking atoms out of solid targets is called 'sputtering'. The other type is 'electronic loss', i.e. the incident ion transfers part of its energy to the electrons. These electrons can either be excited to produce secondary electron emission or be stripped of the atom resulting in the ionisation of atoms and secondary ion emission. The penetration depth (stopping range) of incident ions increases with ion energy and decreases with increasing ion-mass and relative atomic mass of atoms in a solid. Some incident ions will, after losing all their energy, become part of the material (implantation), which can modify the material's local chemical composition. If this is done purposely, it is called 'doping'. Scattering in an amorphous target material is a random process. In crystalline materials, ions can penetrate several times deeper along directions with low Miller indices, as in other directions or amorphous targets. This phenomenon is called the 'ion-channelling effect' and decreases the yield of sputtered target atoms and the emission of secondary electrons. Therefore, sputtering rates on polycrystalline surfaces can be different. Ion-bombardment can also result in amorphisation of the surface layer.

It was revealed that ion-sputtering yield has the following features: The sputtering yield rises with the beam incident angle (with respect to the normal to the sample surface) and reaches maximum at about 80 ° (Orloff, 2003). For gallium ions, the sputtering yield increases with increasing ion-energy only up to 30 keV (yield saturation). The number of re-deposited atoms increases with the depth of the sputtered hole and any decrease in scanning speed. An efficient way to reduce the re-deposition rate is to sputter with a fast multiple scan of the ion-beam. Ion-bombardment combined with chemically-active gases can multiply the sputtering rate. A small amount of a chemically active gas is introduced into a chamber, where the gas molecules adhere to the target surface. Ion-bombardment ionises the gas atoms, which then react with the target-atoms to form volatile compounds which are in turn evacuated by the vacuum system. Appropriate reactive gas must be applied for every target material.

If the gas ions and target atoms produce non-volatile compounds, the reaction products will stay on the target surface. The deposition process and the sputtering process coexist and compete, but the deposition rate can be adjusted to be greater than the sputtering rate. Since deposition only takes place where an incident ion impacts, controlling the ion beam scanning can produce arbitrary shaped 3D structures. Commonly

used gases are organic metal-compound gases, and the deposited materials are organic compounds (containing Ga in the case of gallium ion-source) of Pt, Al, Au or W. Non-metallic materials such as SiO_2 can also be deposited (ORLOFF, 2003).

Sputtering, deposition and doping can be controlled with nanometre-precision. Therefore, FIB is truly a micro-fabrication tool with many applications. In the serial production of IC (integrated circuit) chips, FIB is applied as a diagnostic tool (cross-sectioning of chips for failure analysis), as well as a tool for repairing of IC chips through sputtering or deposition (ZHENG, 2005). FIB implantation can be used to dope the substrates during transistor production. The ability of sputtering is one of the most frequently used features of FIB that can be used directly as a tool for the micro-milling of electronic components, such as read/write heads for modern high density computer hard discs, production of micro-optic switches (XIE, 2003), production of microsurgical manipulators (VASILE, 1999) and for producing micro-milling tools made of steel, diamond or carbides (VASILE, 1999; PICARD, 2003), etc. One of the earliest and still very important applications of FIB systems is preparation (cutting and thinning) of TEM samples for routine inspections during production (electronics), as well as in different fields of science (ZHENG, 2005; PRESSER, 1997; GIANUZZI, 1999; DE VEINEMAN, 1999; SENNHAUSER, 2004). Also in the field of life science the FIB/SEM system provides more comprehensive microscopy results than any conventional microscopy technique in biomedicine (BURKHARDT, 2004; DROBNE, 2004). In dual-beam instruments (FIB/SEM) secondary electron and secondary ion emission caused by ion-bombardment can be used to produce an image providing much more information than conventional SEM-images. In the case of multi-phase samples, but also onephase polycrystalline materials with random orientation of crystal grains, the sputtering rates are not uniform over the scanned area (channelling effect). Although this phenomenon has been referred to as a drawback when preparing a TEM sample or ion-polishing the surface (ORLOFF, 2003) it can also be applied very usefully as an etching technique to reveal the microstructure.

MATERIALS AND METHODS

To investigate the capability of FIB and avoid difficulties pointed out in Introduction, two Cu-Al-Ni alloys were melt-spun: Cu – 13 wt.% Al – 4 wt.% Ni (CuAl13Ni4) and Cu – 15 wt.% Al – 4 wt.% Ni (CuAl15-Ni4). All investigated samples were in as-cast condition. The melt-spun ribbons of alloy CuAl13Ni4 were fully martensitic, whereas the melt-spun ribbons of alloy CuAl15Ni4 consisted of two phases, martensite and γ_2. The presence of the same phases was also determined by (LOJEN, 2005). Some samples of both alloys were mechanically polished (diamond, ¼ μm), whereas the majority of the samples were in as-received condition. The FIB/SEM system used was a FEI Quanta 200 3D equipped with a gallium liquid ion source and a Pt-gas injection system. The following experiments were carried out:

- FIB-etching of diamond-polished cross-sectioned sample, and both the unprepared wheel- and air-side of the

alloy CuAl13Ni4,
- FIB-etching of diamond-polished cross-sectioned sample, and both the unprepared wheel- and air-side of the alloy CuAl15Ni,
- FIB-cutting, -polishing and -etching of the cross-sectioned samples of both alloys.

The polished samples were firstly dismounted from the resin and then attached to the sample holder using a conductive carbon tape. Before placing into the FIB/SEM chamber, all samples were cleaned with alcohol in an ultrasonic cleaner and dried.

All presented micrographs in this work are ion-induced secondary electron images (ISE images). The only exception is Figure 4a, which is an electron-induced secondary electron image (SE image). The presented images were not electronically edited.

Results and discussion

Surfaces and cross-sections

Figure 1a shows the mechanically-polished cross-section of alloy CuAl13Ni4 with six ion-etched fields in the form of squares: dimensions 5 μm × 5 μm. The ribbon air-side of the same alloy was also treated in the same way (Figure 1b). Etching current and etching times are indicated in both figures. It could be seen that the microstructure could not be resolved outside the squares. However, the contrast between microstructural constituents in etched fields increases progressively with etching time. It was clearly revealed that the microstructure is fully martensitic. The contrast between different martensitic plates is caused by the channelling effect, namely, different plates possess different crystallographic orientation. Some of them have low index directions almost parallel to the ion beam; therefore, ions penetrate

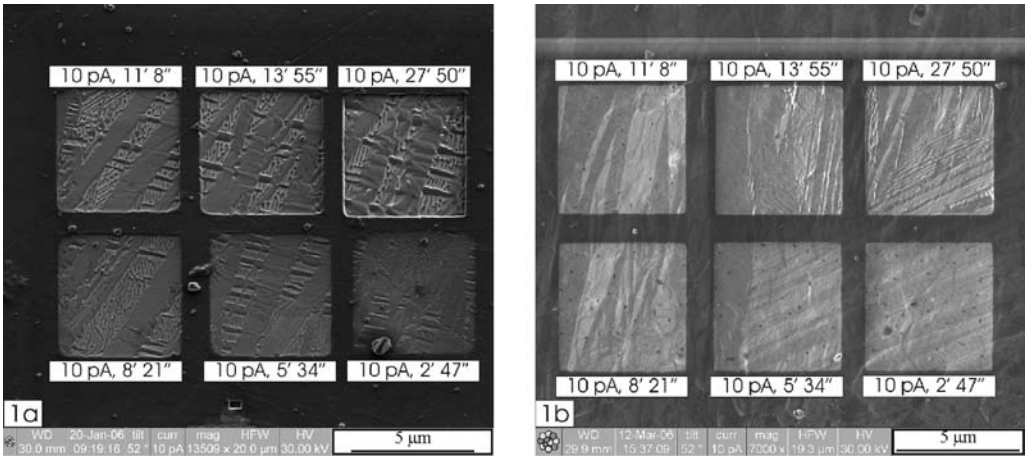

Figure 1. Ion-etched melt-spun ribbon (alloy CuAl13Ni4). a) Mechanically polished cross-sectioned sample; b) as-received air-side of the melt-spun ribbon
Slika 1. Ionsko jedkan hitrostrjen trak zlitine CuAl13Ni4. a) Mehansko poliran prečni prerez, b) metalografsko nepripravljena zunanja površina hitrostrjenega traku

deep into the material. This causes both low scattering yield and low emission of secondary electrons, which finally result in a darker appearance of these plates. It can also be concluded that plates with the same level of greyness have approximately the same orientation to the beam. It seems that in brighter regions the sputtering rate is much higher than elsewhere. In these plates the development of columns can be observed very soon. Such columns are typical FIB-artefacts. Within other plates, which appeared uniform at the beginning of etching, a structure consisting of finer lamellae became visible. By comparing Figure 1a with Figure 1b, it is also obvious that the etching effect is much stronger on the smoother polished sample than on the metallographycally unprepared air-side of the ribbon. Nevertheless, the surface must be clean, without dust or other particles since these particles prevent uniform etching of the surface.

Figure 2 compares images of the same region after mechanical polishing and after 1 hour of FIB-etching with 10 pA (cross-section of a melt-spun ribbon – alloy CuAl15Ni4). As can be expected from Figure 2a, the ribbon consists of two phases, but the contrast is very low. After FIB-etching the two phases can be clearly distinguishable (Figure 2b). It could then be seen that the dendritic γ_2-phase prevails. Two different grey-tones suggest two different crystallographic orientations, and the origin for contrast is the channelling effect. The γ_2-phase on the grain boundary

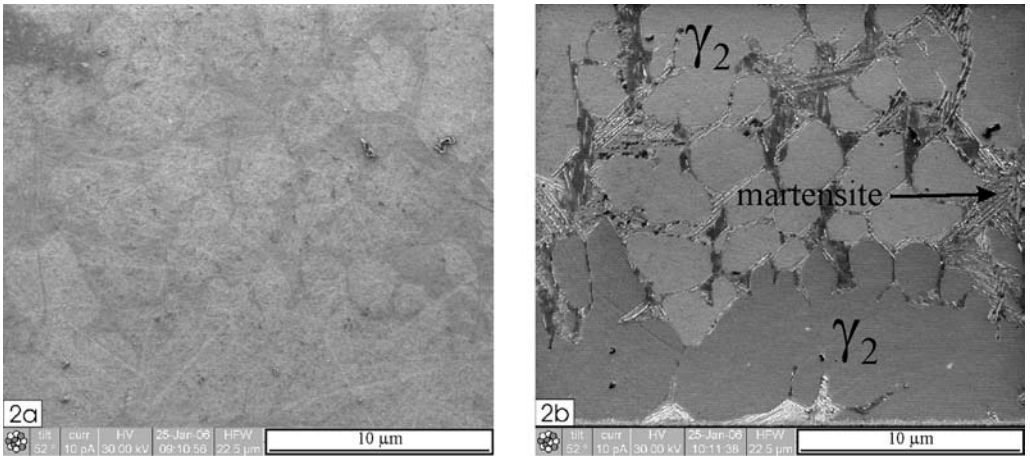

Figure 2. Cross-section of a melt-spun ribbon (alloy CuAl15Ni4). a) ISE-image after mechanical polishing, b) ISE-image of the same area after 1 hour of FIB-etching of the same area, using ion current of 10 pA. Two-phase microstructure is clearly revealed.

Slika 2. Prečni prerez hitrostrjenega traku zlitine CuAl13Ni4. a) Mehansko poliran prečni prerez (slika s sekundarnimi elektroni, ki so jih inducirali ioni), b) mikroposnetek po 1 uri ionskega jedkanja istega mesta s tokom 10 pA (slika s sekundarnimi elektroni, ki so jih inducirali ioni). Dvofazna mikrostruktura je jasno vidna.

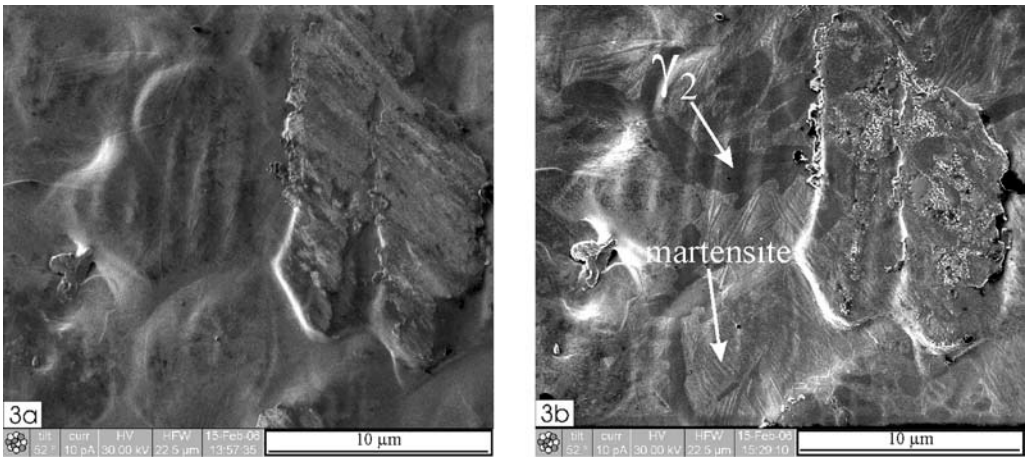

Figure 3. The wheel-side of the melt-spun ribbon (alloy CuAl15Ni4): a) in the as-cast condition; b) after 90 minutes FIB-etching of the same area, using ion current of 10 pA

Slika 3. Površina hitrostrjenega traku, ki je bila pri litju ob kolesu (zlitina CuAl15Ni4): a) v začetnem stanju, b) po 90 minutah ionskega jedkanja istega mesta s tokom 10 pA

appears darker, whilst the intragranular γ_2-phase appears brighter. The characteristic martensitic pattern can be observed in the interdendritic space, consisting of differently-oriented plates.

The unprepared wheel-side surface of the alloy CuAl15Ni4 is shown in Figure 3. The surface is so rough that, besides the surface topography, no microstructural features can be identified (Figure 3a). A comparison with Figure 3b (FIB-etched for 90 min. at 10 pA) makes it obvious that in this case FIB-etching is clearly not as efficient as on smoother surfaces. Although, in Figure 3b, some areas of the martensitic pattern are clearly recognisable and the darker γ_2-phase also distinct, the topographical features cannot be distinguished from the microstructural in many places. It should be stressed that ion-etching means the removal of material by ion-sputtering. Different sputtering rates of differently-oriented grains (plates) and different phases cause the surface to become rough. However, etching is effective when the roughness is only in the order of a few tenths of nanometres. When a larger amount of material is removed, the possibility of introducing different artefacts becomes much more probable. Therefore, if the surface roughness is in the order of a micrometre (Figure 3) than it is obvious that any topographic contrast will be much stronger than the orientational one.

3-D microscopy

Additional freedom during the metallographic preparation of samples allows for so-called 3D-microscopy. In this case a larger portion of material is removed from the surface, which does not require any kind of previous metallographic preparation. We used 3D-microscopy for imaging

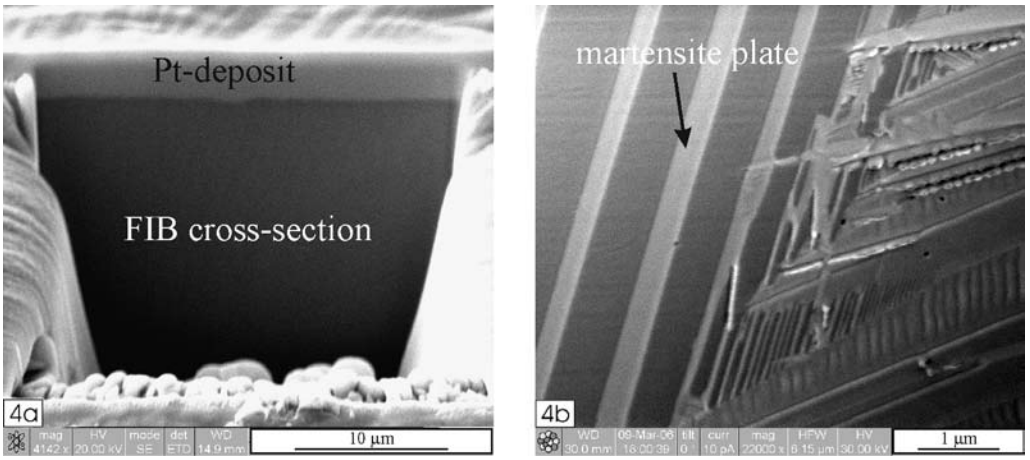

Figure 4. 3D-microscopy of a melt-spun ribbon (CuAl13Ni4 alloy); a) SE image after sputtering and ion-polishing; b) ion induced SE image after 75 min of FIB-etching at 10 pA
Slika 4. 3D-mikroskopija hitrostrjenega traku zlitine CuAl13Ni4; a) posnetek s sekundarnimi elektroni po grobem rezanju in ionskem poliranju površine, b) mikroposnetek po 75 min ionskega jedkanja z ioni (sekundarni elektroni, ki so jih vzbudili ioni)

fully martensitic CuAl13Ni4 alloy (Figure 4). In order to prepare a very smooth cross-section, with a straight and sharp trench edge, a 2 µm thick protective Pt-layer was deposited on the surface (FIB-CVD; FIB chemical vapour deposition). The trench was sputtered with an ion current of 5 nA over two hours. The cross-section surface was then polished with 0.5 nA for an additional hour. The FIB-polished 3-D cross-section is shown in Figure 4a. The brighter layer on the top of the cross-section is the Pt-rich protective layer. The surface below is so smooth, that the contrast of the SE image (Figure 4a is an electron induced SE image) is too poor to reveal the microstructure. On the ion induced SE image the characteristic martensitic pattern can, although with a poor contrast, be recognised without prior FIB-etching (micrograph not shown). After FIB-etching of the cross-section area for 75 minutes with an ion current of 10 pA a contrast rich ion induced SE image can be obtained as shown in Figure 4b.

The 3D cross-section of alloy CuAl15Ni4 was made without a Pt-protective layer. For sputtering the trench and polishing the 3D cross-section shown in Figure 5a the same parameters were selected as in the case of alloy 1 (Figure 4a). When Pt-protection was not used, the free surface surrounding the trench was intensively FIB-etched and the two-phase structure was well revealed (Figure 5a). But in this case the edge of the trench is not as straight as that in Figure 4a and, on the cross-section itself vertical ribs (bright vertical lines in Figure 5b) are present - the so-called 'curtains'. Except for these curtains an indistinctive image of the two-phase structure can already be seen prior to FIB-etching. The phase-contrast increases over the etching-time. The micrograph in Figure 5b was taken after 38

minutes of FIB-etching at 10 pA. However, several artefacts may appear over prolonged etching times.

Conclusions

FIB is an adequate tool for performing the metallographic preparation of melt-spun Cu-Al-Ni ribbons. Most of the difficulties can be avoided when coupled with conventional metallographic preparation. The major disadvantage of in-situ FIB preparation lies in the fact that it is impossible to cut, polish and etch large areas in reasonable time.

According to the results, smaller ion-currents should be preferred for FIB-etching when revealing fine microstructural details. During the preparation of melt-spun Cu-Al-Ni ribbons, a current of 10 pA turned-out to be a good compromise between the revealed details and the necessary etching time for areas within the range 25 μm^2 to 100 μm^2. But, as the etching area increases, the time required to obtain a satisfactory result increases enormously.

It was clearly determined that the efficiency of FIB-etching is higher on smooth surfaces. If the surface was too rough, microstructural features can be overlapped by the surface topography.

In 3D microscopy, application of FIB-deposited Pt-compound protective layer brings advantages as well as disadvantages. If the edge is straight and sharp and the surface of the 3D cross-section smooth, then the protective layer is unavoidable. If applied, the free surface of the sample cannot be seen. However, if we want to observe both the microstructure of the free

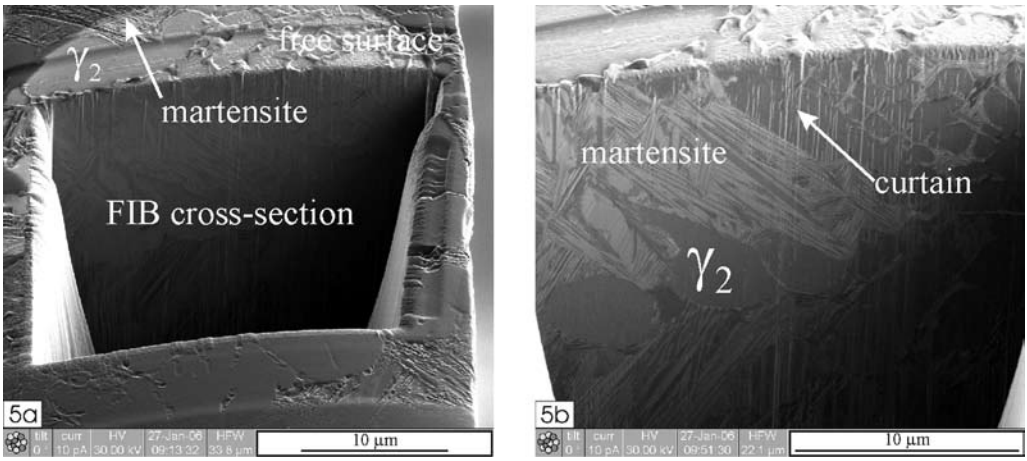

Figure 5. 3D-microscopy of a melt-spun ribbon (CuAl15Ni4 alloy): a) 3D cross-section after sputtering and FIB-polishing; b) After 38 min FIB-etching at 10 pA

Slika 5. 3D-mikroskopija hitrostrjenega traku zlitine CuAl15Ni4. a) 3D-prerez po grobem rezanju in ionskem poliranju, b) mikroposnetek po 38 minutah jedkanja z ioni s tokom 10 pA

surface and the microstructure of the FIB cross-section at the same time, then the presence of FIB-artefacts is almost unavoidable.

POVZETEK

Karakterizacija hitrostrjenih trakov zlitine Cu-Al-Ni z uporabo fokusiranega ionskega curka

Vrstični elektronski mikroskop, ki je opremljen s fokusiranim ionskim curkom (FIB), se uporablja za najrazličnejše namene. Naprava FIB uporablja pospešene galijeve ione za odstranjevanje materiala (mikro- in nanoobdelava), izdelavo prečnih rezov (3D-mikroskopija), pripravo TEM-vzorcev, ionsko mikroskopijo, ipd. Trki težkih galijevih ionov z atomi vzorca sprožijo kaskado najrazličnejših procesov, ki povzročijo razprševanje atomov s površine, implantacijo galijevih atomov v vzorec, nastanek najrazličnejših kristalnih napak, deloma pa lahko postane obstreljevana površina tudi amorfna. V polikristalnih in/ali heterogenih materialih je odstranjevanje površinskih plasti neenakomerno. Hitrosti ionskega jedkanja različnih faz namreč niso enake, poleg tega pa so odvisne od orientacije kristalov. Te značilnosti so lahko koristne pri metalografski pripravi vzorcev. Namreč, neenakomerno razprševanje atomov lahko povzroči primeren kontrast med različnimi fazami in med različno orientiranimi kristalnimi zrni.

Mikrostruktura zlitin z oblikovnim spominom Cu-Al-Ni je lahko po hitrem strjevanju (npr. litju na vrteče kolo) v celoti martenzitna ali pa sestavljena iz dveh faz: martenzita in γ_2. Uporaba standardnih metalografskih postopkov in jedkal lahko povzroči številne težave. Zato smo se odločili, da raziščemo možnosti uporabe FIB pri metalografski pripravi eno- in dvofazne mikrostrukture zlitin Cu-Al-Ni. Glavni cilj je bil optimirati pripravo s FIB izdelanih prečnih prerezov, kot tudi obeh prostih površin hitro strjenih trakov. Ugotovljeno je bilo, da je mogoče z uporabo FIB odkriti mikrostrukturo na vseh površinah, toda najdrobnejše detajle lahko razločimo samo na čistih, mehansko poliranih površinah. Veliki ionski tokovi lahko odkrijejo mikrostrukturo na veliki površini zelo hitro, toda na račun izgube podrobnosti. Zato je potreben kompromis med trajanjem jedkanja, velikostjo raziskovanega področja in ločljivostjo mikrostrukturnih podrobnosti. Najpomembnejši zaključek je: mikrostrukturo hitro strjenih trakov lahko odkrijemo s kombinacijo jedkanja z ioni in 3D-mikroskopijo brez uporabe postopkov klasične metalografske priprave.

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