MINI-THIXOFORMING OF VARIOUS MODIFIED STATES OF THE STOCK STRUCTURE OF TOOL STEEL X210Cr12

MINITIKSO PREOBLIKOVANJE ORODNEGA JEKLA X210Cr12 Z RAZLIČNO ZAČETNO MIKROSTRUKTURO

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Thixoforming is, due to the crossing of the semi-solid state, an alternative method of forming. A forming temperature above the solidus temperature, gives rise to structural components in an arrangement that would be impossible to achieve using other commonly used methods. In the specific area of thixoforming of steels, in comparison to non-ferrous metals, options for appropriate applications are still being explored. The new method is the mini-thixoforming of steels. Unlike the conventional methods of thixoforming, mini-thixoforming achieves very rapid heating gradients and high-speed solidification and cooling. These conditions then significantly influence the development of structure and material properties. This article is focused on describing the selected process parameters and on how they influence the change in the structure from a blank to the resulting product after the processing. The experimental programme was conducted on the X210Cr12 ledeburitic steel; the initial state was modified with different procedures. This tool steel has a high amount of alloying elements and a wide interval between the solidus and liquidus. This makes it suitable for semi-solid-state processing. Two different input states were processed by mini-thixoforming. The first state was the soft-annealed state with a hardness of 211 HV30 and a grain size of approximately 13 µm. The second condition was treated with the severe-plastic-deformation (SPD) method, i.e., with equal-channel angular pressing (ECAP). This led to a grain refinement of less than 1 µm and an increase in the hardness to 370 HV30. For a comparison of the initial and final structures, light and scanning electron microscopies were used, including electron backscatter diffraction (EBSD). To evaluate the mechanical properties a hardness test and a compressive-strength test were selected. The experiment showed the extent to which a microstructural modification of a blank can affect the microstructure obtained by mini-thixoforming, especially in terms of shape change

Tikso preoblikovanje je zaradi prehoda skozi testasto stanje alternativna metoda za preoblikovanje. Temperatura preoblikovanja, ki je nad temperaturo strjevanja, omogoča izdelavo komponent, ki jih ne bi bilo mogoče izdelati po drugih metodah. Za specifično področje tikso preoblikovanja jekel se v primerjavi z neželeznimi kovinami še vedno iščejo opcije za primerno uporabo. Minitikso preoblikovanje jekel je nova metoda. Nasprotno od običajnih metod tikso preoblikovanja minitikso preoblikovanje izkel je nova metoda. Nasprotno od običajnih metod tikso preoblikovanja minitikso preoblikovanje jekel je nova metoda. Nasprotno od običajnih metod tikso preoblikovanja minitikso preoblikovanje izkel ogravanja in veliko hitrost strjevanja in ohlajanja. Te razmere močno vplivajo na razvoj strukture in lastnosti materiala. Ta članek obravnava izbrane procesne parametre in njihov vpliv na strukturo od surovca do strukture preoblikovanca. Eksperimentalno delo je bilo izvršeno pri ledeburitnem jeklu X210Cr12, začetno stanje pa je bilo spremenjeno z različnimi postopki. To orodno jeklo vsebuje veliko količino legirnih elementov in ima širok interval med solidusom in likvidusom. To omogoča, da je jeklo primerno za predelavo v testastem stanju. Z minitikso preoblikovanjem sta bili obdelani dve različni izhodni stanji. Prvo je bilo mehko žarjeno stanje s trdoto 211 HV30 in velikostjo zrn okrog 13 µm. Drugo stanje je bilo močno plastično deformirano z metodo SPD; to je s stiskanjem skozi pravokotni kanal enakih dimenzij (ECAP). To je povzročilo zmanjšanje zrn na manj kot 1 µm in povečalo trdoto na 370 HV30. Za primerjavo začetne in končne mikrostrukture sta bili uporabljeni svetlobna in elektronska mikroskopija, vključno z metodo uklona sipanih elektronov (EBSD). Za oceno mehanskih lastnosti sta bila izbrana preizkusa trdote in tlačne trdnosti. Eksperiment je pokazal, do kakšne mere sprememba mikrostrukture surovca lahko vpliva na mikrostrukturo, dobljeno pri minitikso preoblikovanju, posebno glede spremembe oblike in velikosti grad

Ključne besede: testasto stanje, tikso preoblikovanje, jeklo, minitikso preoblikovanje, ECAP, EBSD, X210Cr12

1 INTRODUCTION

The competition in the market for materials continues to grow, whether it is the increasingly widespread use of plastics, ceramics, composites or any other materials. However, despite these alternatives steel has an irreplaceable position among construction materials due to its properties. It holds this position thanks to the new methods of processing, enhancement of the existing conventional production technologies, and the development of new processing procedures. These procedures may also include semi-solid-state processing, such thixoforming. During thixoforming the material is processed in a semi-solid state, which, on the one hand, causes technological problems with the heating and manipulation of the blank.^{1,2} On the other hand, this condition enables the creation of a dimensionally precise and complex product and makes a full use of the chemical potential of both common and less accessible medium- and high-alloy steels. The variant of thixoforming called mini-thixo-forming, developed for very small volumes of a material and the products with a small size, gave rise to the structural components of the microstructure of a material B. MAŠEK et al.: MINI-THIXOFORMING OF VARIOUS MODIFIED STATES OF THE STOCK STRUCTURE ...



Figure 1: X210Cr12 steel microstructure in the soft-annealed state **Slika 1:** Mikrostruktura jekla X210Cr12 v mehko žarjenem stanju

that are usually not obtained with other technologies. At the same time, it is possible to form a product of a complex shape with the dimensions in the order of millimeters.

2 EXPERIMENTAL PROGRAM

2.1 Experiment

Ledeburitic high-chromium steel X210Cr12 was chosen as the experimental material, primarily used for the production of tools for cold forming (**Table 1**). The material was standardly formed and soft annealed after casting. For the structure with low optical clarity, an EBSD analysis was used. Primary chromium carbides of $M_{23}C_7$ and secondary carbides were easy to distinguish in the structure of the material, but clearly distinguishable high-angle grain boundaries of the ferritic grains were missing (**Figure 1**). The measured hardness was 211 HV30. For the processing in the semi-solid state, the proportion of the liquid phase should be in the range between 40–60 %.^{1,2} The X210Cr12 interval for thixoforming is relatively wide, about 60 °C.^{3,4} Nevertheless, it is



Figure 2: Channel with the bending angle of $\Phi = 120^{\circ 5}$ **Slika 2:** Kanal s kotom upogiba $\Phi = 120^{\circ 5}$

necessary to precisely control the temperature profile throughout the process. After solving this technical problem, a high amount of alloying elements gave rise to a number of unconventionally arranged structural components, significantly influencing the structure. The research of the transmission of structural attributes between the initial and final structures of the modified microstructural states was carried out. One research method was ECAP.

Table 1: Chemical composition of X210Cr12 steel in mass fractions, w/%

Tabela 1: Kemijska sestava jekla X210Cr12 v masnih deležih, w/%

С	Cr	Mn	Si
1.8-2.05	11-12.5	0.2-0.45	0.2-0.45

2.2 Modification of the structure with ECAP

ECAP was used to modify the original structure even though ledeburitic steel has a relatively high strength and it is difficult to form. To use this method of intense deformation, a cylindrical steel sample with the dimensions of d = 7 mm, l = 50 mm was molded into a prism of austenitic steel. After a gradual optimization, a blank with the cross-section of $10 \text{ mm} \times 10 \text{ mm}$ and a length of 120 mm was created. These dimensions matched the dimensions of the channel in the ECAP device. This procedure was chosen, in particular, to prevent damage to the instrument and a blockage or seizure of the sample during extrusion. The prism was then expelled through the channel with a bending angle of $\Phi = 120^{\circ}$ (Figure 2).⁵ Between one and four passages with the rotation by 90° (Route B) and 180° (Route C) were carried out. For the experiment four passes with the rotation of 180° (Figure 3) were finally chosen.⁶ The process was carried out at the elevated temperature of T = 350 °C. The rate of passage was 10 mm s⁻¹, the force applied was F = 310 kN with the back pressure. After the deformation of the encapsulated blank, the case was removed, the structure was analyzed and the semi-products for mini-thixoforming were prepared. EBSD analysis was performed to



Figure 3: ECAP schema, shear plane passes through the channel, route C, first and second pressings^6

Slika 3: Shematski prikaz ECAP, ravnine striženja gredo skozi kanal, pot C, prvo in drugo stiskanje 6

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Figure 4: Schematic cross-section of the sample Slika 4: Shematski prikaz prereza preizkušanca

evaluate the microstructure after ECAP for the same reasons as in the case of the annealed state.

2.3 Semi-solid processing

Semi-products with a diameter of 6 mm and a length of 46 mm were made from the annealed material and from the material after ECAP. They were processed in the semi-solid state with an experimental device developed for mini-thixoforming.⁷ It uses a combination of high-frequency induction and resistance heating for the blank. This allows precise control of the temperature and deformation during high-speed heating and cooling. The samples were first treated with free tamping without using a mould. Tamping heads also served as electrodes for direct heating of the stock (**Figure 4**).⁷⁻¹⁰ On the basis of an indicative calculation from JMatPro¹¹ a processing temperature of 1265 °C was selected. The heating was carried out from *RT* to the forming temperature in 8 s, followed by holding 1 s at this temperature and rapid cooling to *RT* in 2 s.

2.4 Structural analysis

In order to compare the influence of the as-received annealed structure with the resulting structure after semi-solid processing, the samples were analyzed with light and scanning electron microscopies. The samples were prepared with etching using V₂A (10 mL HNO₃, 0.3 Vogels Sparbeize, 100 mL HCl, 100 mL H₂O). Mechanical properties were determined by measuring the hardness in HV30 and by testing the compressive strength of the cylindrical samples with a diameter of 5 mm and a length of 5 mm, while the strain rate during the test was 1 s⁻¹.



Figure 5: EBSD analysis of: a) the annealed state and b) modified state after ECAP **Slika 5:** EBSD-analiza: a) v žarjenem stanju in b) spremenjeno stanje po ECAP



Figure 6: a) Band contrast of the annealed state and b) modified state after ECAP **Slika 6:** a) Žarjeno stanje in b) spremenjeno stanje po ECAP

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3 RESULTS AND DISCUSSION

The material in the annealed state had a grain size of 13.5 µm. By modifying the annealed state with intense deformation using ECAP, the expected structure refinement was achieved. The grain size was 0.75 µm (Figures 5, 6 and 7). The analysis showed that the microstructures of the two prepared states were sufficiently homogeneous on the entire monitored sections. The effect of the deformation on the primary chromium carbides is clearly discernible in the structure (Figure 8). The hardness increased from 211 HV30 to 377 HV30. As regards the mechanical properties, the processing in the semi-solid state led to an increase in the hardness. Comparing the annealed state and the state after ECAP, the hardness value increased from (211 resp. 377) HV30 to (350 resp. 382) HV30. The yield strength $R_{p0.2}$ determined with the compressive-strength test of the sample in the annealed state increased after the semi-solid processing from 415 MPa to 800 MPa. A structure consisting of polyhedral M-A components and a eutectic lamellar network was obtained using this process. M-A components contained metastable austenite and martensite. Twins could be observed in the structure in both cases. Due to the high heating temperature a complete dissolution of the large primary chromium carbides occurred. These formed the first lamellas of the eutectic network during cooling. Small primary chromium carbides were incompletely or partially dissolved in the grains of the M-A components. Those remaining on the polished surface after etching produce the relief. An incomplete dissolution of carbides is attributed to the short heating time. The difference between the structures obtained from the annealed



Figure 7: Results of the EBSD analysis: a) an evaluation of the grain size of the annealed state and b) the state after refinement using the ECAP process

Slika 7: Rezultati EBSD-analize: a) ocena velikosti zrn v žarjenem stanju in b) po udrobnjenju z uporabo ECAP-postopka



Figure 8: Microstructure of X210Cr12 steel: a) in the longitudinal section, b) in the annealed state and c) the state after ECAP **Slika 8:** Mikrostruktura jekla X210Cr12: a) vzdolžni prerez, b) žarjeno stanje in c) stanje po ECAP

condition and the state after ECAP is especially apparent in the distribution of individual structural components. In the state prepared using ECAP a change in the regularity and in the orientation of the eutectic network is easily observed; compared to the annealed state, this state does not have such linear, regularly oriented areas, showing a less regular character (**Figure 9**). At the same time the eutectic network is distributed on the polyhedral grain boundaries and does not create coarser groups.

Also, the size and shape of the grains are slightly different. The ECAP modified structure is more regular

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Figure 9: Structure after processing in the semi-solid state by mini-thixoforming: a) state of the annealed blank and b) state after ECAP; LM, polarized light, V_2A etched

Slika 9: Struktura po predelavi v testastem stanju z minitikso preoblikovanjem: a) žarjeni surovec in b) stanje po ECAP-predelavi, svetlobna mikroskopija, polarizirana svetloba, jedkalo V_2A

in the shape of the polyhedral bodies and contains a higher proportion of smaller grains (**Figure 9**).

This corresponds to the microstructure obtained after ECAP, where large primary chromium carbides are, in addition to the fragmentation and the total fragmented area, distributed from their original positions parallel to each other in the longitudinal direction, with a hint of linearity, taken up after the treatment of the primary



Figure 10: Characteristic arrangement of primary carbides and the eutectic network in the states before and after ECAP and semi-solid processing

Slika 10: Značilna razporeditev primarnih karbidov in evtektične mreže pred ECAP-predelavo in po njej ter po predelavi v testastem stanju

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production (Figure 10). This resulted in an insignificant increase in the hardness compared to the semi-solid processing of the annealed state.

4 CONCLUSION

Two states of ledeburitic tool steel X210Cr12 were processed using mini-thixoforming. They were the soft-annealed state and the state refined with ECAP. When using ECAP large primary chromium carbides were fragmented and the structure was refined. At the same time, a partial distortion of the linearity and a shift of carbides due to deformation in the transverse direction were achieved. With the semi-solid processing of the two prepared microstructural states, a microstructure consisting of polyhedral bodies of the metastable austenite containing M-A compounds was achieved. These polyhedral bodies were embedded in a eutectic network. As regards the distribution and shape of the polyhedral bodies, it was possible to observe the mutual differences between the annealed state and the state modified with ECAP. After the semi-solid processing of the annealed state, eutectic formations were oriented in parallel, forming rather large clusters. On the other hand, in the structure obtained by ECAP processing the ledeburiticeutectic network was more regular and embedded between single austenitic grains. The shape of the polyhedral grains of M-A compounds was more regular for the ECAP semi-product. The yield strength $R_{p0.2}$ established by compressive-strength testing in the annealed state increased with the processing in the semi-solid state from 415 MPa to 800 MPa.

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