EFFECT OF THE INITIAL MICROSTRUCTURE ON THE PROPERTIES OF LOW-ALLOYED STEEL UPON MINI-THIXOFORMING

VPLIV ZAČETNE MIKROSTRUKTURE NA LASTNOSTI MALOLEGIRANEGA JEKLA PO PREDELAVI MINI-THIXOFORMING

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Thixoforming is an unconventional forming process based on semi-solid processing. Semi-solid processing involves heating the feedstock to a temperature at which it becomes partly liquid and partly solid. Thanks to partial melting and rapid solidification, unconventional microstructures can be obtained even in conventional materials. Today's research efforts mainly involve high-alloyed steels with a wide freezing range at lower temperatures. By contrast, low-alloyed steels with the solidification temperatures above 1400 °C are not under extensive investigation.

The main objective of the study was to find whether and how the attributes of the feedstock microstructure can be transmitted to the final semi-solid processed microstructure of the 30MnVS6 microalloyed steel. The first stage of the experiment involved mini-thixoforming of the as-received specimens of the steel. In order to assess the effects of the initial microstructure, specimens with three types of microstructure were prepared with high-pressure torsion (HPT), a severe-plastic-deformation (SPD) technique. Initial and final microstructures were examined using light and electron microscopes and image-analysis techniques. The phase composition was determined with the aid of the X-ray diffraction analysis. The mechanical properties were determined with tensile and hardness testing. Information on the local properties and the properties of microstructure constituents was obtained with microhardness measurement.

Keywords: minithixoforming, 30MnVS6, severe plastic deformation, high pressure torsion

Thixoforming je neobičajen postopek preoblikovanja, ki temelji na preoblikovanju v testastem stanju. Preoblikovanje v testastem stanju vključuje ogrevanje preoblikovanca do temperature, pri kateri postane material delno v staljenem in delno v strjenem stanju. Po zaslugi delnega taljenja in hitrega strjevanja se lahko doseže neobičajne mikrostrukture celo pri navadnih materialih. Sedanje raziskave se izvajajo večinoma na močno legiranih jeklih s širokim intervalom strjevanja pri nižjih temperaturah. Nasprotno se malolegirana jekla s temperature strjevanja nad 1400 °C ne preiskuje intenzivno.

Glavni cilj te študije je ugotoviti, ali in kako atribute mikrostrukture surovca prenesti po preoblikovanju v testastem stanju pri mikrolegiranem jeklu 30MnVS6. Prva stopnja eksperimentov je vključevala minithixoforming preoblikovanje dobavljenih vzorcev jekla. Za oceno vpliva začetne mikrostrukture so bili pripravljeni vzorci s tremi vrstami mikrostrukture s torzijo pri visokem tlaku (HPT), to je s tehniko velike plastične deformacije (SPD). Začetne in končne mikrostrukture so bile preiskane s svetlobno mikroskopijo, z elektronsko mikroskopijo in s tehnikami analize slik.

Sestava faz je bila določena z rentgensko difrakcijsko analizo. Mehanske lastnosti so bile določene z nateznim preizkusom in z merjenjem trdote. Informacija o lokalnih lastnostih in lastnostih posameznih mikrostrukturnih faz je bila dobljena z meritvami mikrotrdote.

Ključne besede: minithixoforming, 30MnVS6, plastična deformacija, torzija pri visokem tlaku

1 INTRODUCTION

Mini-thixoforming was developed for processing small volumes of a material using highly dynamic heating and cooling processes.^{1,2} Thanks to these characteristics, the processing induces transformations that are not commonly encountered in the conventional processes. As a consequence, various types of microstructure are obtained which contain metastable constituents, such as austenite in ferritic-pearlitic steel.³ Owing to the highly dynamic nature of the phenomena involved, the initial condition of a material cannot be neglected because the material's history is often reflected in its final microstructure. This fact frequently fails to be acknowledged and has not been explored adequately. The objective of the present experiment was to compare the character of the resulting microstructure with the

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initial condition. The method used to modify the microstructures was a severe-plastic-deformation technique known as high-pressure torsion $(HPT)^4$ and it involved various amounts of the applied strain.

2 EXPERIMENTAL PROGRAMME

The experimental programme was conducted with the 30MnVS6 microalloyed steel (**Table 1**). This steel, alloyed predominantly with manganese and silicon, is typically used for making forged parts. The as-received material was in the form of rolled and peeled normalized bars. The as-received microstructure consisted of ferrite and pearlite (**Figure 1**). The ultimate and yield strengths in the as-received condition were 770 MPa and 570 MPa, respectively, and the hardness was 237 HV10.

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Table 1: Chemical composition of 30MnVS6 steel in mass fractions (w/%)	
Tabela 1: Kemijska sestava jekla 30MnVS6 v masnih deležih (w/%)	

С	Mn	Si	Р	S	Cu	Cr	Ni	Al	N	Mo	V	Ti
0.31	1.50	0.62	0.018	0.024	0.03	0.20	0.02	0.02	0.0122	0.007	0.098	0.0261

With regard to the low content of alloying elements, a tentative simulation using the JMatPro program was used to ascertain that the freezing range of the material was relatively narrow and lying at high temperatures.⁵ The temperature interval was 1425–1490 °C. The temperature to be used for achieving the semi-solid state region was set at 1455 °C.

2.1 Refinement of the initial microstructure using HPT

The structure of the material was refined using the HPT method, one of the severe-plastic-deformation techniques. Its principle is a torsional deformation between stationary and rotating dies that compress the material (**Figure 2**).⁶ The magnitude of the strain introduced depends on the number of revolutions. The feedstock was a disc with a 35 mm diameter. The forming process reduced its height to 7.5 mm and expanded its diameter to 38 mm (**Figure 3**). The forming was performed at ambient temperature. The material did not undergo any recrystallization. The effect of the initial microstructure



Figure 1: As-received microstructure of 30MnVS6 steel Slika 1: Mikrostruktura jekla 30MnVS6 v dobavljenem stanju



Figure 2: Schematic of the HPT process⁶ **Slika 2:** Shematski prikaz HPT-postopka⁶



Figure 3: Final products of the HPT process **Slika 3:** Končni proizvod HPT-postopka

was explored using three specimens upon various numbers of revolutions: 3.5, 4.25 and 7.

2.2 Microstructure after HPT

As the amounts of the strain in the centre and by the edge of the formed disc differ, the metallographic section was prepared on the disc face. The observations with both the light and electron microscopes confirmed that the resulting microstructure was heavily distorted, in contrast to the as-received material. The most severe distortion was found in the specimen upon the highest number of revolutions, which was seven (Figure 4a). Its microstructure still contained the ferrite and pearlite mixture but both phases were heavily deformed (Figure 4b). Its hardness, up to 502 HV10, was higher than that of the as-received material. The surprising finding was that the pearlite lamellae underwent a heavy deformation without any fracturing. Another interesting feature was



Figure 4: a) Microstructure of 30MnVS6 steel upon HPT processing, 7 revolutions,;) detail of distorted pearlite lamellae Slika 4: a) Mikrostruktura jekla 30MnVS6 po HPT-predelavi, 7 vrtljajev, b) detajl izkrivljene lamele perlita

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Figure 5: Schematic of the workpiece manufacturing sequence **Slika 5:** Shematski prikaz korakov priprave obdelovanca



Figure 6: Final workpiece welded together with an electron beam Slika 6: Končna oblika obdelovanca, zvarjenega z elektronskim curkom

that upon three or even four revolutions, the microstructures in the centre of a disc and by its edge showed little difference. This was evidenced by similar hardness levels for all the areas of the deformed disc. Upon 3.5 revolutions, the hardness was 421 HV10. Upon 4.25 revolutions, it rose to 465 HV10.

2.3 Preparation of the workpieces for semi-solid processing

The non-uniform strain intensity was reflected in the not quite uniform microstructures of the discs upon HPT. The difference was most notable between the outermost edge and the centre of a disc. For this reason, the edge and the centre of a disc were not used for further experiments. Cylinders of a 15 mm height were cut from the discs. This dimension is consistent with the length of the active part of the workpiece for mini-thixoforming. The remaining conical parts of the workpiece were made for low-carbon steel with a high melting temperature. The entire workpiece for semi-solid processing was thus assembled from three parts and electron-welded in a vacuum (Figure 5). This low-energy welding method left the special microstructure in the active part of the workpiece unaffected. The workpieces from the as-received material were made in one piece. Their length was 48 mm and their diameter was 6 mm (Figure 6). The purpose of their conical ends is to carry the electric current for heating and to centre the workpieces inside the die.

3 SEMI-SOLID PROCESSING

All the workpieces were formed in a titanium die with a groove-shaped cavity with a 20 mm length and a 5 mm \times 1.9 mm cross-section. This die was specially



Figure 7: Schematic illustration of the whole process with the microstructure development after individual steps **Slika 7:** Shematski prikaz celotnega postopka z razvojem mikrostrukture po vsakem koraku

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developed for the mini-thixoforming process.⁷ With respect to the high heating temperature, an R-type thermocouple (rhodium-platinum) was used. The heating temperature was found using stepwise optimization. Despite the temperature being high, it was maintained accurately, as was the heating rate (**Figure 7**).

The initial experiments were conducted with the as-received specimens. Only after all the mini-thixo-forming parameters were determined, the HPT-processed specimens were employed. First, a suitable heating temperature was sought. A total of three temperatures were tried: (1450, 1455 and 1460) °C. The heating time was 61 s. The deformation applied within 0.3 s was followed by rapid solidification enhanced by the contact with the metal cavity surface. The initial cooling rate reached 300 °C/s.

4 DISCUSSION OF RESULTS

At the first stage, the semi-solid processing parameters were optimized for the 30MnVS6 steel employed outside its typical domain. Mini-thixoforming of the as-received non-refined material produced a martensitic microstructure with some amount of bainite. This is unusual, considering the typical thixo-formed microstructure, which consists of polyhedral austenite grains embedded in a ledeburite network. In the specimen heated to 1460 °C, the etchant used for outlining prior austenite grains (the picric acid) revealed that the majority of the product and the interior of the workpiece had a dendritic morphology. In response to this finding, the heating temperature was reduced. However, with the temperature reduced by 10 °C, the cavity was not filled completely because the liquid fraction was inadequate.

The most suitable heating temperature proved to be 1455 °C. Once the processing parameters were optimized, the product filled the die cavity completely. The microstructure in the centre of the product contained mostly martensite and some bainite (**Figures 8a** and **8b**). Its hardness was approximately 767 HV10. The etchant for the prior austenite grain boundaries revealed that the proportion of the dendrites in the product decreased.



Figure 8: Micrographs of the as-received material upon semi-solid processing: a) workpiece centre, b) centre of the extruded product Slika 8: Mikrostruktura dobavljenega materiala po predelavi v testastem stanju: a) sredina obdelovanca, b) sredina vzorca po ekstruziji

Besides the dendrites, there were polygonal shapes of the prior austenite grains in the microstructure. These shapes were found predominantly in the product formed by forcing the semi-liquid material into the groove in the titanium die. A higher proportion of bainite was found in the workpiece interior, whose cooling rate was lower than that of the product.

Processing the workpieces made from the discs upon 3.5 revolutions led to a similar character of the microstructure. It consisted of a mixture of martensite and bainite. The hardness in the centre of the product reached no more than 500 HV10. The variation in the microstructure was only found after outlining the prior austenite grain boundaries by etching, whereas the normally etched martensitic-bainitic microstructure appeared uniform. The morphology of the prior austenite grains in the mini-thixoformed HPT-refined material was polygonal. In contrast to the as-received material, no large dendritic areas were found upon mini-thixoforming (Figures 9a and 9b). Etching the material processed with HPT with 4.25 revolutions and picric acid revealed areas with very fine globular particles with the size of approximately 20 µm (Figure 9c). The matrix consisted of martensite and bainite, as in the previous cases. The hardness in the centre of the product was 713 HV10. No effects of a further refinement were found in the specimens upon 7 revolutions.



Figure 9: Comparison between the morphologies of prior austenite grains upon semi-solid processing of the material with two different initial microstructures: a) micrograph of as-received material, b) micrograph of HPT-refined material, c) detail of fine globular grains **Slika 9:** Primerjava videza prvotnih avstenitnih zrn po predelavi v testastem stanju materiala z dvema različnima začetnima mikrostrukturama: a) mikrostruktura dobavljenega materiala, b) mikrostruktura materiala po HPT-postopku, c) detajl drobnih globulitnih zrn

5 CONCLUSION

The effect of the initial microstructure of the microalloyed 30MnVS6 steel on its final microstructure upon semi-solid processing was explored. The material in the two initial states was mini-thixoformed under identical conditions. The first state was characterized by a ferrite-pearlite microstructure with the hardness of 237 HV10 and the other was a plastically deformed microstructure caused by high-pressure torsion. The hardness level was between 421 and 502 HV10, depending on the strain intensity.

All the products made with mini-thixoforming contained martensite with a small fraction of bainite. The final hardness was high, reaching 713 HV10 in some cases. The refinement of the initial microstructure was not reflected substantially in the semi-solid processed microstructure. After the heaviest deformation, the workpiece material had a very fine morphology of the prior austenite grains. In order to characterize them, additional microscopic techniques will have to be used such as EBSD to find their size or X-ray diffraction analysis to describe their texture.

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