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GRAIN-REFINING ABILITY OF ULTRA-RAPID ANNEALING FOR LOW-CARBON STEEL: SEVERE PLASTIC DEFORMATION

SPOSOBNOST UDROBNENJA ZRN PRI ZELO HITREM ŽARJENJU MALOOGLJIČNEGA JEKLA PO VELIKI PLASTIČNI DEFORMACIJI

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To study the grain-refinement ability of ultra-rapid annealing (URA), heating rates from 0.3 °C/s up to 1200 °C/s with conventional annealing and URA in the intercritical temperature range were performed on severely deformed low-carbon steel. The results show that recrystallization in conventional annealing is completed below the critical temperature of Ac_1 without grain refinement. URA up to 730 °C at a heating rate of 200 °C/s causes grain refinement due to full interaction between the recrystallization and phase transformation. URAs up to 730 °C with heating rates of 600 °C/s and 1000 °C/s lead to partial grain refinement and no grain refinement, respectively. During annealing with a heating rate of 1200 °C/s, the temperature should reach 760 °C for the occurrence of recrystallization leading to grain refinement. The sample URAed at 600 °C/s up to 730 °C shows the maximum hardness due to fine grains being partially formed.

Keywords: conventional annealing, ultra-rapid annealing, interaction, severe plastic deformation

Za študij sposobnosti udrobnjenja zrn pri zelo hitrem žarjenju, so bile uporabljene hitrosti segrevanja od 0,3 °C/s do 1200 °C/s pri običajnem in zelo hitrem žarjenju v interkritičnem temperaturnem območju na močno deformiranem maloogljičnem jeklu. Rezultati kažejo, da je rekristalizacija pri konvencionalnem žarjenju zaključena pod kritično temperaturo Ac_1 brez udrobnjenja zrn. Zelo hitro žarjenje do 730 °C pri hitrosti ogrevanja 200 °C/s povzroči udrobnjenje zrn, zaradi polne interakcije med rekristalizacijo in fazno premeno. Zelo hitro žarjenje do 730 °C , s hitrostjo ogrevanja 600 °C/s in 1000 °C/s povzroči delno udrobnjenje zrn oz. udrobnjenja zrn ni. Med ogrevanjem s hitrostjo 1200 °C/s je potrebno doseči temperaturo 760 °C, da se pojavi rekristalizacija, ki povzroči udrobnitev zrn. Vzorec po zelo hitrem žarjenju z 600 °C/s do 730 °C kaže maksimalno trdoto zaradi delnega nastanka drobnih zrn.

Ključne besede: konvencionalno žarjenje, zelo hitro žarjenje, interakcija, močna plastična deformacija

1 INTRODUCTION

Conventional annealing of deformed steel with a slow heating rate (usually below 10 °C/s)[1] causes recovery and recrystallization to occur sequentially by subcritical temperature (below the critical temperature of Ac_1).[2,3] This annealing is followed by furnace soaking to complete the recrystallization.[3] However, in low-carbon steel the grain refinement after conventional annealing needs to combine the steel's chemical composition and the annealing cycle.[4,5] Also, the phase transformation during conventional annealing in the intercritical temperature range does not influence the grain refinement of deformed low-carbon steel.

On the other hand, ultra-rapid annealing (URA) of steels is a new method that leads to grain refinement in a very short time due to changes in the starting temperature of recrystallization.[6–9] This condition provides the ability to control the microstructure by means of the simultaneous occurrence of recrystallization and phase transformation. URA shifts the start temperature of recrystallization to higher values near and above Ac_1.[3,7–15] So, in the intercritical temperature range, recrystallization and phase transformation proceed concurrently and with a mutual "interaction". The intensity of the interaction depends of the amount of stored energy and determines the final microstructure of the steel.[16–18] The heating rate controls the recrystallization start temperature and the amount of stored strain energy before the interaction. So, heating rate has a key role in controlling the microstructure and the grain refinement.

In this research, the effect of a slow heating rate (0.3 °C/s) up to a high heating rate (1200 °C/s) in the intercritical temperature range on the final microstructure of the severely deformed low-carbon steel is examined. The steel is subjected to severe plastic deformation to increase its stored strain energy before annealing.

2 EXPERIMENTAL PART

The low-carbon steel with 0.05 C, 0.2 Mn, 0.011 P, 0.007 S, 0.055 Al (all in *w*/%) in the form of a sheet with a thickness of 3 mm is used in this research. The as-received materials were normalized to obtain the spherodized cementite. SPD was performed using two passes of constrained grooved pressing (CGP) on specimens with a size of 80×50×3 mm in the sheet rolling direction. Each complete pass of CGP imposes an overall strain of 1.16, which contains four alternating stages of pressing grooving in dies and flattening dies. After the first two stages, the specimen is rotated 180° around the axis perpendicular to the sheet plane and then the two stages are repeated. The direction of the grooving dies is perpendicular to the rolling direction of the as-received sheet. Teflon layers were used as the lubricant between the samples and the dies. More details of the CGP method are given in [19,20]. For ultra-rapid annealing, the samples were machined from both sides up to a thickness of 1.8 mm, and then cut into specimens with a width of 14 mm.

The URA setup is based on resistance heating, which is provided by a high current transformer controlled by a digital control unit. A high current of about 5000 A passes through the sample and increases the temperature that is measured using a thermocouple finely spot welded to the center of the sample, and read with a high-speed data logger at a frequency of 3000. The data is concurrently transferred and saved to a PC, and the temperature-time diagram is drawn simultaneously during URA. Different heating rates were applied up to the desired sample temperature that was then immediately cooled after heating with a rate of about –700 °C/s with a high-speed spray of water and air.

Slow heating rate conventional annealing was carried out in a furnace with a heating rate of about 0.3 °C/s without any soaking time. At the desired temperature the samples were removed from the furnace and quenched as mentioned above.

After annealing, a hardness test was performed using 30 kg Vickers and then the samples were prepared for microstructural examinations, from thickness in the longitudinal direction.

3 RESULTS AND DISCUSSION

The microstructure of the as-received steel consists of ferrite grains (a mean grain size of about 80 μm) with dispersed carbide particles and was free of pearlite. After SPD, the mean grain size was reduced to about 30 μm, and particles were refined and highly dispersed at the grain boundaries and inside the grains (**Figure 1**).

The microstructure of the SPDed steel after conventional furnace annealing up to 730 °C (slightly above Ac_1) is shown in **Figure 2**. Several experiments were done by annealing up to different temperatures showed that the A_{C1} temperature of the studied steel is about 720 °C and pearlite was observed after heating higher than 720 °C. The microstructure was recrystallized, the grains grew and some of the grains are marked with "A", grown to abnormal size. This shows that recrystallization started before the critical temperature of Ac_1. **Figure 3** shows the microstructures of SPDed steel after subcritical conventional annealing up to the temperatures

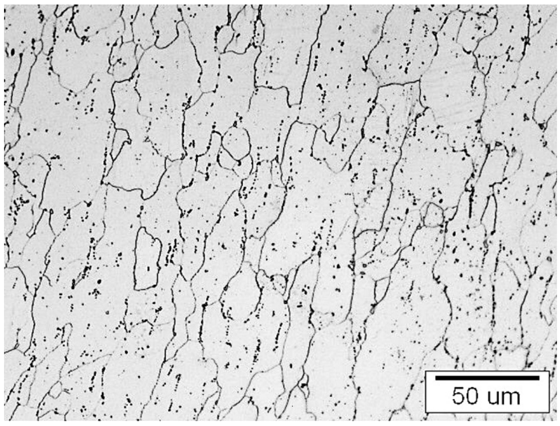

Figure 1: Microstructure of low-carbon steel after SPD
Slika 1: Mikrostruktura malooglјičnega jekla po SPD

of 500 °C and 650 °C. After heating up to 500 °C, the microstructure was similar to that of the SPDed sample and indicated that the recrystallization was not started. In the sample heated to 650 °C, the microstructure is fully recrystallized and the grain size is smaller than in **Figure 2**. This shows that the start and finish temperatures of the recrystallization during conventional annealing are much below the Ac_1.

The microstructure of SPDed steel after URA with a heating rate of 200 °C/s up to 730 °C in **Figure 4** showed that URA produced grain refinement to an average grain size of 8 μm.

The scanning electron micrograph (SEM) in **Figure 4b** indicates the formation of pearlite massively dispersed at recrystallized ferrite grain boundaries. This suggests the full interaction of the ferrite recrystallization and the phase transformation. As the interaction occurs, the kinetics of recrystallization and transformation are increased.[3,6,13,16,21] The austenite phase nucleates first at the deformed ferrite grain boundaries.[22] On the other hand, austenite provides a preferred recrystal-

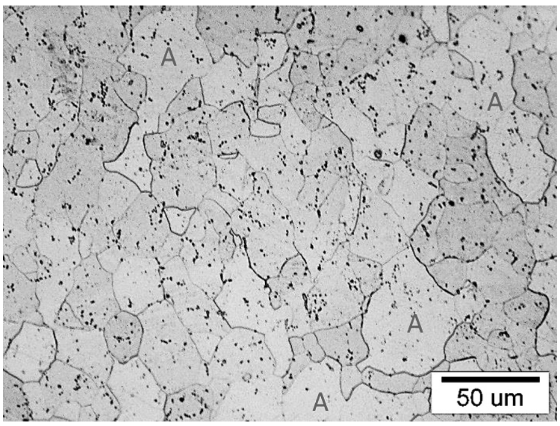

Figure 2: Microstructure of severely deformed steel by furnace annealing up to 730 °C with heating rate of 0.3 °C/s, abnormally grown grains are marked with A
Slika 2: Mikrostruktura močno deformiranega jekla po žarjenju v peči na 730 °C in s hitrostjo segrevanja 0,3 °C /s, neobičajno velika zrna so označena z A

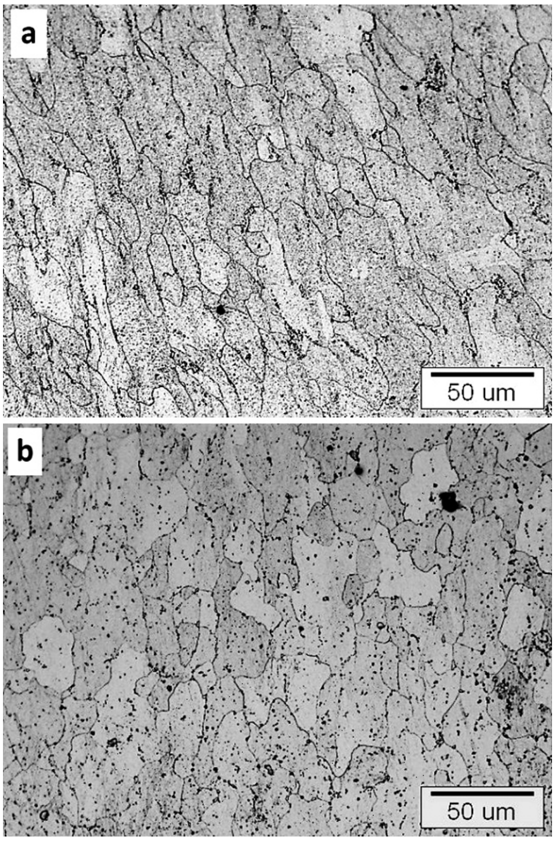

Figure 3: Microstructure of severely deformed steel after furnace annealing with a heating rate of 0.3 °C/s: a) 500 °C, and b) 650 °C

Slika 3: Mikrostruktura močno deformiranega jekla po žarjenju v peči s hitrostjo segrevanja 0,3 °C/s: a) 500 °C in b) 650 °C

lization nucleation site for the non-recrystallized ferrite. So, fine recrystallized ferrite grains are surrounded by austenite phases (which are transformed to pearlite during cooling) and their growth is inhibited and the microstructure with refined grains is formed. It should be noted that the transformation of austenite to pearlite during cooling was accelerated by the small size of the austenite grains.[12,23,24]

URA with a heating rate of 600 °C/s up to 730 °C does not show a homogeneous grain refinement. This

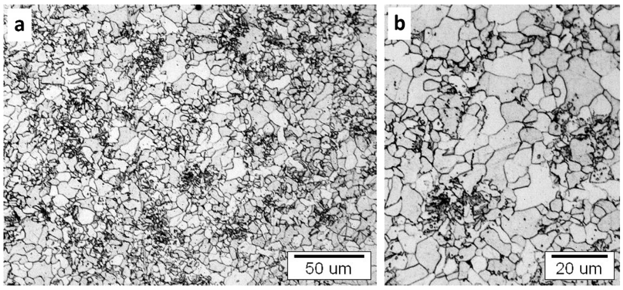

Figure 5: a) Microstructure of severely deformed steel after URA up to 730 °C with heating rate of 600 °C/s, b) higher magnification of a)

Slika 5: a) Mikrostruktura močno deformiranega jekla po zelo hitrem žarjenju do 730 °C s hitrostjo segrevanja 600 °C/s in b) večja povečava a)

microstructure (**Figure 5**) consists of two-sized grains of 10 µm and 2 µm. It can be seen that a partial grain refinement occurred due to incomplete recrystallization, and pearlite surrounded by the locally recrystallized ferrite grains were formed.

This shows a localized interaction. In comparison with the sample heated with 200 °C/s, the heating rate of 600 °C/s delays the start temperature of the recrystallization slightly above Ac_1. During URA, austenite was formed and then recrystallization took place. In this situation the interaction between ferrite recrystallization and the formation of austenite is localized around recently formed austenite.

The microstructure shown in **Figure 6** is from the SPDed sample URA treated with heating at 1000 °C/s up to 730 °C and shows no recrystallization and the grain size of about 30 µm is similar to the grain size of the SPDed sample before URA. A very high heating rate of 1000 °C/s shifts the start temperature of the recrystallization higher than 730 °C. However, the transformation occurred and the pearlite phase was formed around the non-recrystallized ferrite grains (**Figure 6b**).

For the occurrence of recrystallization with a high heating rate, the peak temperature of URA should be increased in proportion to the heating rate. For example, from the microstructure in **Figure 7**, for URA with

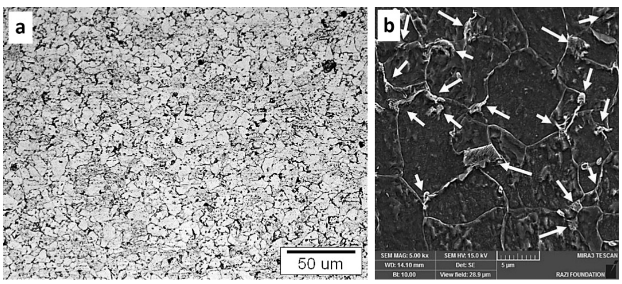

Figure 4: Microstructure of severely deformed steel after URA up to 730 °C with heating rate of 200 °C/s: a) optical microscopy image, and b) SEM image, arrows mark pearlites

Slika 4: Mikrostruktura močno deformiranega jekla po zelo hitrem žarjenju na 730 °C, s hitrostjo segrevanja 200 °C/s: a) posnetek s svetlobno mikroskopijo in b) SEM-posnetek, puščice označujejo perlite

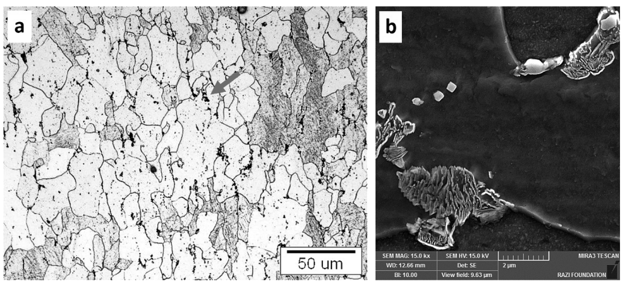

Figure 6: Microstructure of severely deformed steel after URA up to 730 °C with the heating rate of 1000 °C/s: a) optical microscopy image, b) SEM image of dark phases indicated by arrow in a)

Slika 6: Mikrostruktura močno deformiranega jekla po zelo hitrem žarjenju na 730 °C, s hitrostjo segrevanja 1000 °C/s: a) posnetek s svetlobno mikroskopijo in b) SEM-posnetek temnih faz v a), označeni so s puščico

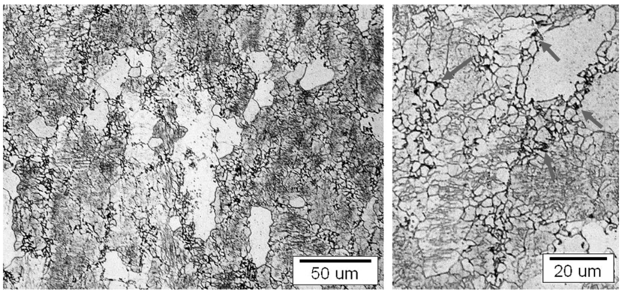

Figure 7: a) Microstructure of severely deformed steel after URA up to 760 °C with heating rate of 1200 °C/s, b) higher magnification of a), arrows marks the pearlite

Slika 7: a) Mikrostruktura močno deformiranega jekla po zelo hitrem žarjenju na 760 °C, s hitrostjo segrevanja 1200 °C/s, b) večja povečava a), puščice označujejo perlit

heating rate of 1200 °C/s up to 760 °C it is clear that there is the start of recrystallization.

In this condition, similar to **Figure 5**, transformation is started before recrystallization. During URA, the austenite phases were formed and then the recrystallization localized around them, causing the formation of the two-sized-grains microstructure with the sizes of 30 µm and 2 µm. The austenite transformed to pearlite during cooling is marked with arrows in **Figure 7b**.

The hardness of the specimens treated conventionally and with URA are shown in **Figure 8**.

The sample heated with 600 °C/s up to 730 °C has the highest hardness because of the partial formation of fine grains and the formation of pearlite in the microstructure. The composite-like microstructure of this sample is responsible for its high hardness. For a similar reason, the sample heated with 1200 °C/s up to 760 °C also has a relatively high value of hardness of about 183 HV.

The hardness of the conventionally annealed samples confirms the described microstructure. The sample annealed up to 550 °C has a similar hardness to that before annealing. This confirms the absence of recrystallization, as mentioned before. For the samples annealed up to 650 °C, the hardness is sharply reduced due to recrystallization.

4 CONCLUSIONS

The effect of the heating rate on the grain refinement is investigated with conventional annealing and ultra-rapid annealing for severely deformed low-carbon steel and the following conclusions can be drawn:

- In conventional annealing with a heating rate of 0.3 °C/s, recrystallization is completed below the critical temperature of Ac_1, and the absence of grain refinement is confirmed with hardness values.
- URA with the heating rate of 200 °C/s up to 730 °C leads to full grain refinement due to the full interaction between ferrite recrystallization and austenite formation.
- At 730 °C, achieved with URA, the increasing of the heating rate to 600 °C/s causes a partial grain refinement due to localized interaction. The sample in this condition shows the maximum hardness value of 205 Hv.
- By increasing the heating rate to 1000 °C/s, no recrystallization occurs at the temperature of 730 °C.
- Temperature should be increased in proportion to the heating rate in order to start the recrystallization and the occurrence of grain refinement, such as those that occurred for the sample URAed with 1200 °C/s up to 760 °C.

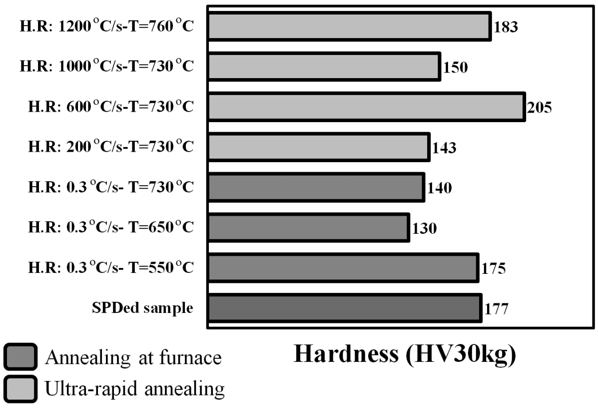

Figure 8: Hardness values of SPDed steel before annealing and after conventional and ultra-rapid annealing.

Slika 8: Vrednosti za trdoto SPD jekla pred žarjenjem in po konvencionalnem ter po zelo hitrem žarjenju

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