THE EFFECT OF NEUTRON IRRADIATION ON MECHANICAL PROPERTIES OF CuCrZr ALLOYS PROCESSED BY CRYOROLLING

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Abstract

Research and development of construction materials for fusion reactors is an important international task. CuCrZr material is a promising material for fabrication of coolant tubes inside the divertor and other components. In recent decade, materials with ultrafine-grained (UFG) structure are widely used due to their unique physical and mechanical properties. To be used in fusion technology, the material of the coolant tube should withstand pressure, show sufficient corrosion properties and neutron irradiation resistance. The UFG materials are known to improve the strength, ductility, heat and corrosion resistance, and according to the recent studies, they are expected to provide improved irradiation resistance. The aim of this study is to investigate the cryorolled UFG CuCrZr material behavior after neutron irradiation and to relate it to the microstructural changes. The post-irradiation evaluation was performed in Hot-cells facilities in Research Centre Řež. Mechanical properties were tested by static tensile test at ambient temperature, supplemented by microstructural analysis using SEM-EBSD. The results were compared with material processed by convention rolling method at ambient temperature.

Keywords: CuCrZr alloy, cryorolling, neutron irradiation, mechanical properties, post-irradiation evaluation

1. INTRODUCTION

The materials to be used in the fusion energy systems must withstand high temperatures and should fulfill certain parameters. The materials for high heat flux applications must provide high thermal conductivity, good thermal stability, strength and tensile ductility, fracture toughness, fatigue and creep–fatigue, and radiation resistance [1-4]. Materials with sufficient properties can be used in fusion reactor for the transfer of a large amount of heat generated inside plasma. According to the recent studies, copper alloys are suitable for example for the heat transfer components. Copper alloys acquire their good thermal transfer properties by precipitation hardening or secondary phases in the material. The most preferable precipitation hardened copper alloy is CuCrZr, which chemical composition is about 1% Cr and 0,1% Zr. This alloy is designed for International Thermonuclear Experimental Reactor (ITER) and is referred to as CuCrZr-IG, with the chromium content of 0.60-0.90 wt.% and of 0.07-0.15 wt.% zirconium [5]. Increased strength of this alloy can be mainly achieved by precipitation hardening of fine Cu_nZr precipitates, which also leads to a considerable improvement of ductility at elevated temperatures [6-7]. Precipitation of Cr-rich particles during supersaturated solid solution aging at 450–480°C for 2–4 h also results in strengthening. It was shown that the neutron irradiation of CuCrZr alloy at the temperatures below 473 K leads to the degradation of mechanical properties, such as a significant increase in tensile strength and severe loss of work hardening ability and uniform elongation. [8-9]. In case of life extension of this alloy under severe conditions of radiation, the microstructure modification is required in order to increase the plasticity and thermal stability of the alloy. This can be achieved using the severe plastic deformation (SPD), which is a very effective method of production of bulk ultrafine - grained (UFG) and nanostructured materials with novel characteristics [2,7]. The mechanical properties and microstructure of CuCrZr alloy can be modified by several SPD methods, such as high-pressure torsion (HPT), multi-directional forging (MDF), equal-channel angular pressing (ECAP) and asymmetric rolling (ASR), in some cases also at cryogenic temperatures [7,10–13]. The increased ductility was observed in Cu-10%Al with UFG structure compared to the conventional coarse-grained alloy [14]. In materials with low stacking fault energy (SFE), SPD processes are realized under cryogenic conditions when the deformation by twinning is activated, which ensures the stability of the grain boundaries at elevated temperatures.

The aim of this study is to investigate the effect of low-dose neutron irradiation (0,1 dpa – displacement per atom) on the mechanical properties of ASR CuCrZr alloy prepared at room temperature and under cryogenic conditions.

2. MATERIALS AND METHODS

Five millimeters thick Cu-0.96% Cr-0.06% Zr alloy plate was used in this study. The specimens were annealed at 1020 °C for 30 min and water quenched prior to the experimental asymmetric rolling. Experimental asymmetric rolling was carried out using rolls with different diameters with the ratio of 2.4. The samples were rolled to achieve 60% total thickness reduction at the end of the cycle. The samples were rolled under the same deformation conditions at room temperature (ASaR) and at cryogenic temperature (AScR). Cryogenic conditions were secured by immersing the samples into liquid nitrogen before and after rolling process for 5 minutes. After experimental rolling, the precipitation heat treatment was carried out at 430 °C for 30 min followed by air cooling. The artificial aging temperature of this UFG microstructure was determined based on the results of our previous research [13].

The set of prepared samples was irradiated in a nuclear research reactor LVR-15 in Research Center Řež, Czechia. The neutron irradiation was performed at a maximum temperature of 230 °C for 61 days to reach total radiation damage of 0.1 dpa. During irradiation, samples were stored in aluminum case, separated by graphite paper. After irradiation, samples were transported to the hot-cells facility in Research Centre Řež, where the post-irradiation examination (PIE) was carried out.

The flat tensile (FT) specimens were cut from the plate in the longitudinal direction of rolling (RD). Specimens used for mechanical testing and direction of rolling are shown in **Figure 1**. Static tensile testing was performed using universal servo-hydraulic tensile testing machine Instron 8874. Constant extension rate testing with 1 mm/min rate was performed up to rupture at room temperature (RT) before and after irradiation. The elongation was measured using video extensometer from the gauge section of the FT specimens. The yield stress (YS) and ultimate tensile strength (UTS) of ASaR and AScR CuCrZr alloy before and after neutron irradiation were obtained.

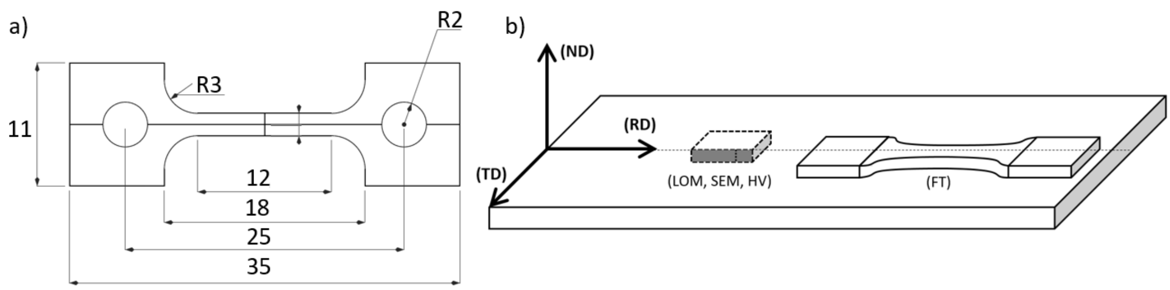

Figure 1 Flat tensile specimen a) and its orientation relative to rolling direction b)

Sample for light optical (LOM), scanning electron microscopy (SEM) and microhardness measurements were prepared by standard Struers methods for copper alloys. LOM was carried out by using the Zeiss Axio Vert.A1 microscope. SEM Tescan MIRA 3 installed in the hot cell was used for microstructure analysis.

The SEM Mira 3 with auto-emission cathode Field Emission Gun (FEG) working with acceleration voltage 0.05 - 30 kV equipped with the electron backscatter diffraction (EBSD) Nordlys Max3 detector operating at 15 kV was used.

The Vickers microhardness (HV 0.1) was measured on the polished surface of the cross-section in transversal direction (**Figure 1b)**). The Struers Duramin 5 device was used for the profile measurement along the thickness of the 1 mm specimen cross-section with the 0,1 mm step.

3. RESULTS AND ANALYSIS

Microstructure of CuCrZr after 60% ASaR and AScR and heat treatment (HT) in rolling direction (RD) is shown in **Figure 2**. Microstructure after deformation and solution annealing is strongly bimodal. After rolling at RT, the deformed elongated grains in RD are observed. The shear bands are present in several grains. After rolling at cryogenic temperature, the deformed grains with high shear bands density are observed. The change in the strengthening mechanism is also apparent from the results of microhardness measurement shown in **Table 1** and **Figure 3**. The HV0,1 increases after ASaR to 140,6 and up to 160,6 after HT. The further increase is observed after AScR, after which HV0,1 is comparable to the ASaR+HT - 166,2. The highest HV was detected in the AScR materials after HT. The microhardness does not vary throughout the cross-section. In addition, no significant difference in microstructure after precipitation strengthening was observed compared to deformed material (**Figure 3**).

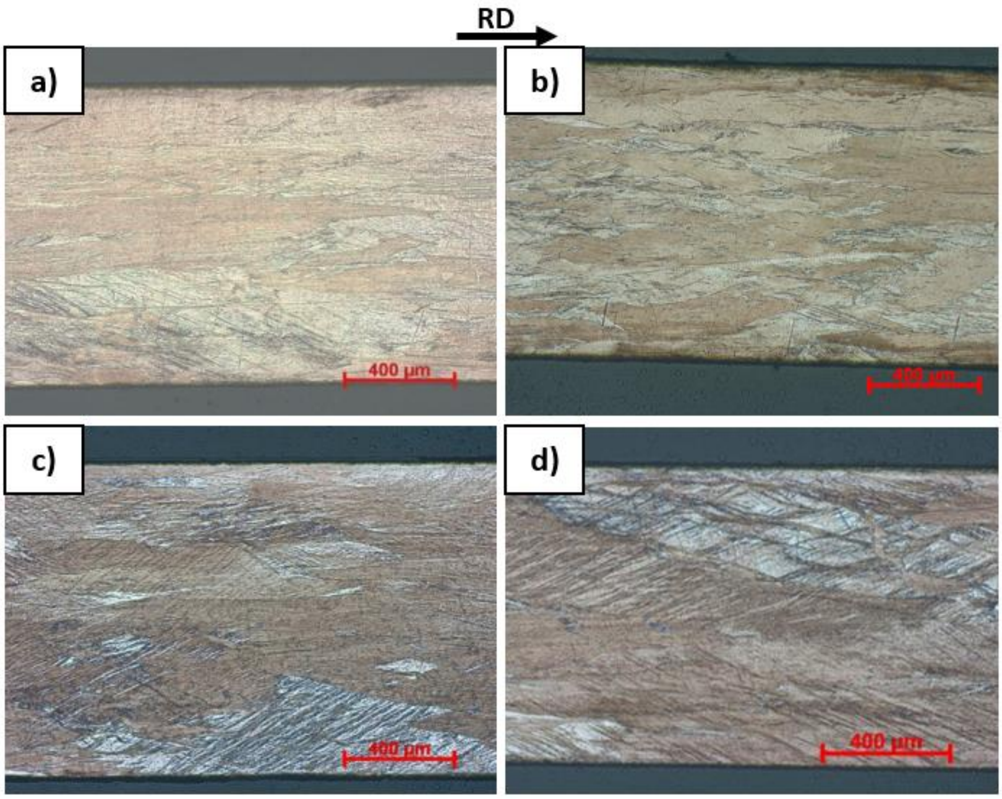

Figure 2 Microstructure (LOM): a) 60% ASaR; b) 60% ASaR + HT; c) 60% AScR; d) 60% AScR + HT

Table 1 Hardness average values of individual states of the material after 60% deformation

Material condition	Initial state	ASaR	ASaR +HT	AScR	AScR +HT
HV 0,1	60,2	140,6	160,6	166,2	202,7

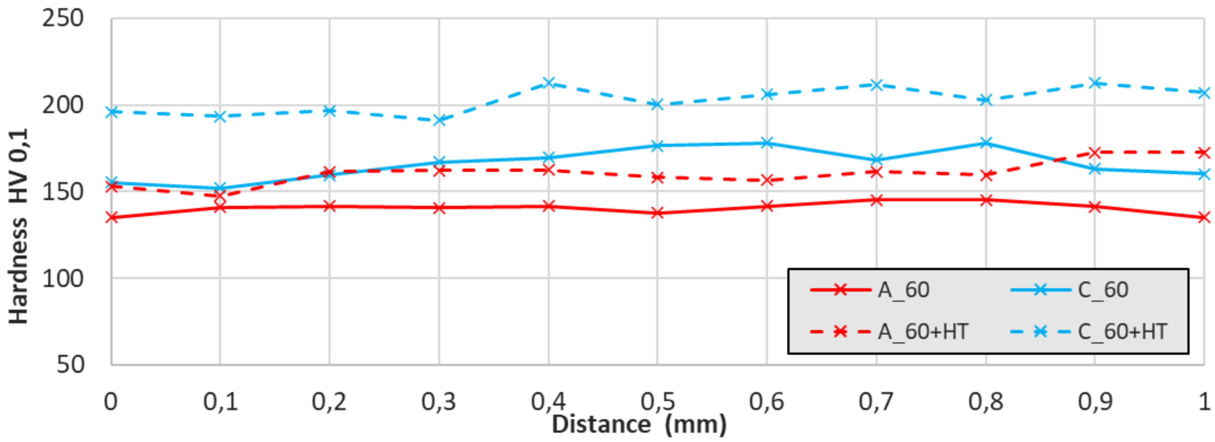

Figure 3 The hardness profile of the thickness of the material

In **Figure 4** inverse pole figures for 60 % ASaR and AScR 60 % are shown. The EBSD analysis has confirmed a higher proportion of shear bands after rolling, especially in the middle area of the sample due to the asymmetric nature of the process. The inhomogeneous deformation of the grains occurs due to different diameters of the rollers. A different mechanism of plastic deformation takes place in case of AScR – twinning occurs in the initial stage. The proportion of twin grain boundaries in the case of ASaR is 0.121% while in the case of AScR it is 1.95% in an analyzed area of 800x600 µm.

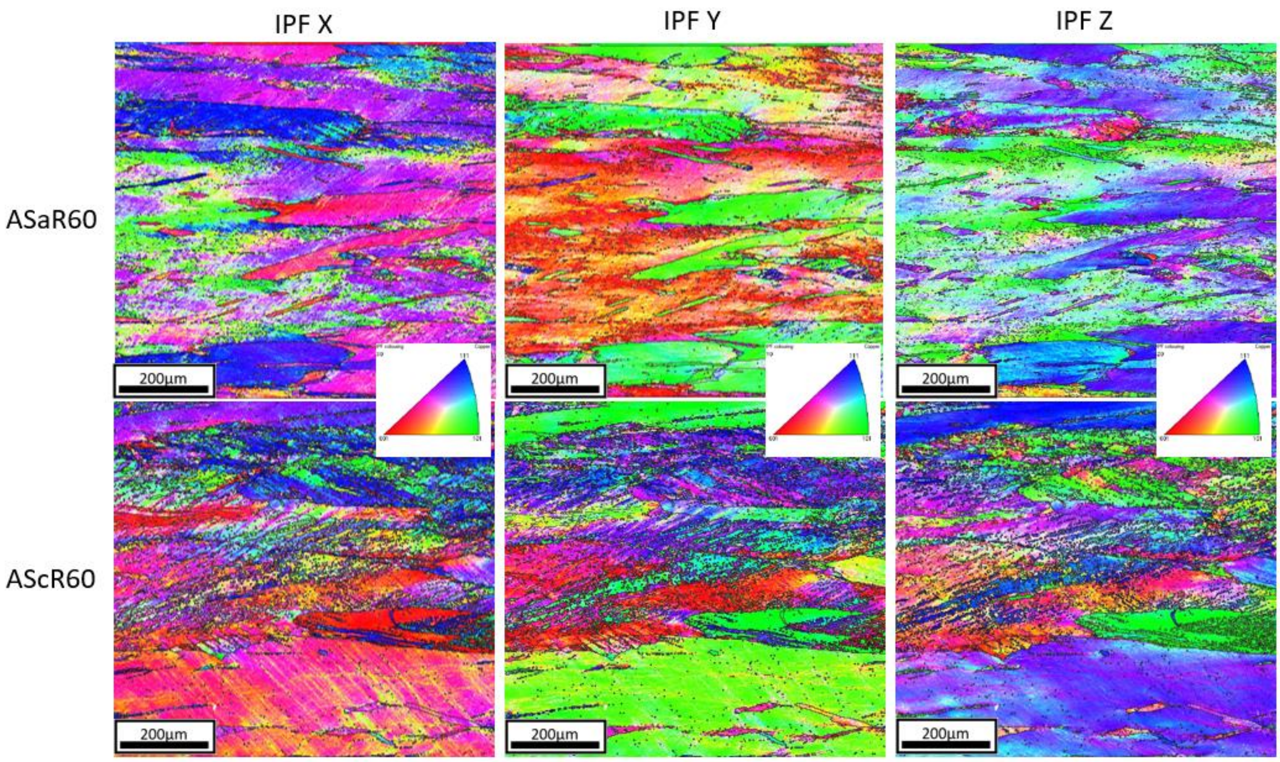

Figure 4 Inverse pole figures of ASaR 60%

Stress-stress curves of ASaR material rolled at RT and after HT before and after neutron irradiation are shown in **Figure 5a)**. The curve shape for CuCrZr rolled at RT is a typical for the highly deformed structures, where instabilities in plastic region are present during uniaxial deformation. These instabilities are caused by the presence of large amount of dislocation in the initial structure and inability to absorb newly created dislocations for uniform strengthening. Moreover, the necking occurs in the initial phase of the deformation process. Material after ASaR has high YS of 340 MPa and the UTS of 346 MPa. However, it shows significantly low uniform elongation of 0,5 % and a ductility of 6,8 %. ASaR CuCrZr after HT has different trend of the stress-strain curve. The higher YS of 376 MPa and the UTS of 410 MPa are observed as well as the uniform elongation of 4,5%. The change of the UTS is caused by the precipitation strengthening while the favorable increase of ductility up to 11% is due to the recovery of initially deformed structure. The necking region is 6,5 %, which does not differ from the deformed state. Stress-strain curves of both ASaR and ASaR+HT after neutron irradiation (0.1 dpa) are shown in **Figure 5a)** using dashed lines. The irradiation lead to the increase of the YS and UTS of 176 MPa and 173 MPa, respectively, and a slight decrease of uniform deformation of 0,4% and ductility of 4,5%. The more pronounced effect of irradiation can be seen in HT material, where the increase of the YS is by 229 MPa and ΔUTS by 206 MPa. At the same time, the uniform deformation decreases by 0,3 % as well as elongation, by 5%.

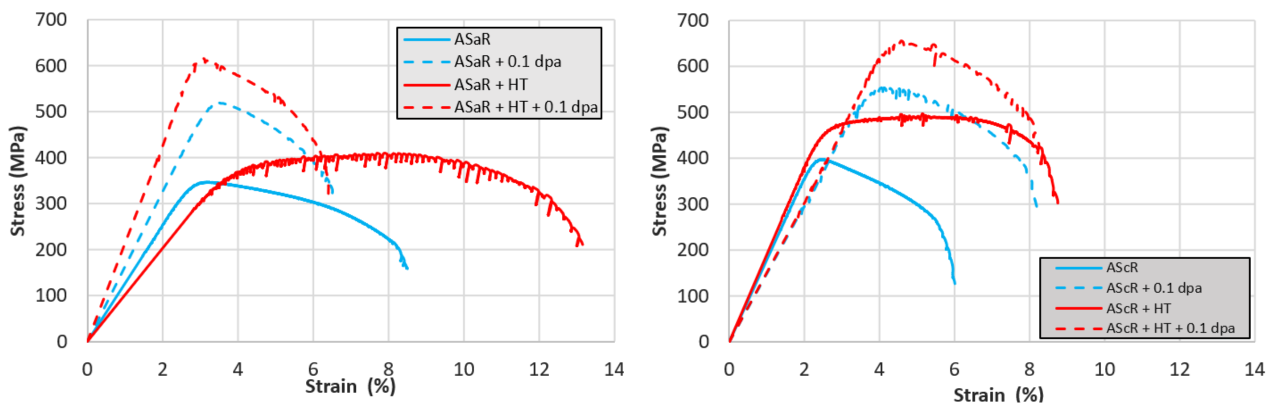

Figure 5 Stress–strain curves for CuCrZr alloy: a) after ASaR and ASaR with HT before and after neutron irradiation, b) after AScR and AScR with HT before and after irradiation

The stress-strain curves of CuCrZr rolled in cryogenic condition at 77K are shown in **Figure 5b)**. The mechanical properties of AScR improve compared to the ASaR. UTS is higher for ASaR by 50MPa after rolling and by 85MPa after rolling and HT. The character of deformation is similar as can be seen from the comparison of stress—strain curves in **Figure 5**. The higher UTS and YS is caused by the suppression of dynamic recovery during deformation under cryogenic condition and the activation of twinning. The latter is observed in the microstructure inside the shear bands in a form of nanotwins, as it was reported in [12,13]. After irradiation, both AScR and AScR + HT are deformed uniformly up to 0,5%, while ductility is 6% and 5,5 %, respectively. It indicates a slight increase of ductility compared to the copper alloy rolled at RT. The irradiation leads to the increase of the YS and UTS for AScR of 132 MPa and 158 MPa and for AScR + HT of 175 MPa and 158 MPa, respectively.

4. CONCLUSION

This study is focused on the evolution of mechanical and structural properties of CuCrZr alloy precipitation hardened by asymmetric rolling at RT and in liquid nitrogen with subsequent neutron irradiation. The following conclusions can be drawn from the experimental results:

- After 60% deformation at RT, the microstructure is considerably deformed, while during deformation in liquid nitrogen the structure is formed by a high amount of shear bands. Presence of shear bands were confirmed by EBSD.

- The presence of shear bands was also reflected in the values of microhardness, which increased by about 25 HV0.1 compared to ASaR. Their presence is noticeable also after precipitation hardening, when the hardness reached 202.7 HV 0.1.
- The values of mechanical properties from the static tensile test have the same character as the values of microhardness. The highest values were reached after AScR + HT where YS = 460 MPa and UTS = 490 MPa.
- Radiation damage of 0.1 dpa caused a significant increase in YS and UTS. Lower values were observed in CuCrZr alloy after AScR.

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