

The Effect of Strain Rate and Initial Grain Size on Deformation Behavior of OFHC Copper at Elevated Temperatures

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Received: 14 October 2021

Accepted: 27 May 2022

DOI: 10.18466/cbayarfbe.1009553

Abstract

Understanding of plastic deformation mechanisms and/or microstructural changes of metals and alloys at elevated temperatures makes possible to control their hot working behavior and final mechanical properties. The aim of the present work is to optimize the conditions to achieve maximum ductility in terms of initial grain size, process temperature and deformation rate. In this study, the OFHC (oxygen-free high conductivity) copper samples of different initial grain sizes (25, 50, 100 and 150 μm) were subjected to tensile tests at temperatures 300, 405, 500 and 700 $^{\circ}\text{C}$ ($0.42 - 0.75 T_m$) and cross-head speeds of 1, 2, 5, 10, 20 and 50 mm/min (strain rates of $5.6 \times 10^{-4} - 2.8 \times 10^{-2} \text{ s}^{-1}$). Experimental results indicated that particular conditions (initial grain size of 50 μm ; 700 $^{\circ}\text{C}$ of working temperature and $5.6 \times 10^{-3} \text{ s}^{-1}$ of strain rate) should be provided in terms of process temperature and deformation rate depending upon initial grain size for dynamic recrystallization and also maximum ductility.

Keywords: OFHC copper; strain rate; elevated temperature; tensile test

1. Introduction

Various types of forming methods of metallic materials are used in technology and material acquires some properties depending on the applied method. Processes such as rolling, deep drawing, extrusion, pressing, forging in the manufacturing of flat products, wires and pipes are carried out in cold or hot conditions. Since cold forming processes of metallic materials are limited, hot forming processes are applied in a wide variety. Hot forming also homogenizes the material and provides reaching the final desired properties by controlling the microstructure [1-6]. Therefore, it should be known how the behavior at the elevated temperatures of the materials is dependent on the parameters such as material microstructure, working temperature and strain rate. This information helps us increasing the production speed and obtaining both higher ductility and strength in the product during the production step [7-8].

Hot forming is the oldest method before forming by casting in the history of technology. Research on archeological findings showed that natural copper was formed with primitive tools by forging in the hot conditions in Anatolia since Acheramic Neolithic times (BC 8000) [9, 10].

High temperature generally decreases material's strength and increases the ductility. Weakening of the bonds among the atoms at elevated temperatures and thus increasing the possibility of movements of the atoms and movement of dislocations by the possibility of substitution especially the possibility of the climbing movement are the reasons for this situation. Atomic vacancies formed at the elevated temperatures also accelerate the diffusion. In addition, slip systems can change by temperature and this eases the deformation. Slip occurs at the grain boundaries due to weakening of the grain boundaries at high temperatures. On the other hand, other diffusion-controlled phenomena can also happen and microstructure changes [11-15].

A lot of research has been made for understanding the complex deformation behavior of the materials at elevated temperatures and studies continue with a great acceleration at present. Various mechanisms were claimed regarding the behavior of the materials at elevated temperatures, mechanism maps were prepared and empirical relationships were developed. Research results put forward that different deformation mechanisms occur at certain temperature and strain rate intervals at high temperature deformations such as hot working, creep and superplastic deformation [16-23].

In the recent years, it has been observed that some fluctuations occurred in the tensile diagrams of some important engineering materials and ductility increased. It has been detected that this was related to the dynamic recrystallization and dynamic grain growth [24-32]. The critical strain for dynamic recrystallization depends on the composition of the material, grain size prior to deformation and deformation temperature and strain rate [33,34].

The purpose of this study is to optimize the conditions to reach at the maximum ductility in the OFHC copper due to the requirements for determination of load during the forming processes, estimation of the metal flow pattern to represent the plastic flow behavior of the material from the point of view of initial grain size, working temperature and strain rate.

2. Materials and Methods

In this study, cold drawn OFHC copper rods (in the purity of 99.99%) were used to investigate the effect of initial grain size, strain rate and deformation temperature on elevated temperature deformation behavior.

High temperature tensile tests were performed at elevated temperatures to investigate the hot working behavior and the final mechanical properties of OFHC copper. High temperature tensile test rods were prepared from cold drawn copper rods in the original diameter of 10 mm by lathe. Tensile test rods had initial grain size of 25 μm and were annealed for 0.5 hours at the temperatures of 300, 700 and 900 $^{\circ}\text{C}$ to obtain

specimens with initial grain sizes of 50, 100 and 150 μm respectively. The specimens were etched by the solution including 5 g of FeCl_3 and 95 ml of methanol and 2 ml of HCl (concentrated) after metallographic polishing methods. Initial grain sizes of the copper specimens were measured by an image analysis method. For this purpose, linear interception method was used excluding annealing twins and 10 mm^2 of area were considered on the specimens.

The surfaces of the high temperature tensile test rods were ground by 1200 grit sandpaper and smooth and clear surfaces were obtained. High temperature tensile tests were performed at the temperatures of 300, 405, 500, 700 and 750 $^{\circ}\text{C}$, and at the strain rates of 1, 2, 5, 10, 20, 50 mm/min by the tensile testing machine (Instron 1195, High Wycombe, England) under atmospheric conditions. Temperatures and strain rates were determined by considering the ductility values obtained from the tests which were carried out to examine the region where optimum results were obtained in more detail. Test temperatures correspond to absolute temperature values of 573 - 1023 $^{\circ}\text{K}$ ($0.42 - 0.75 T/T_m$) and cross-head speeds correspond engineering strain rates of $0.5 - 30 \times 10^{-3} \text{ s}^{-1}$. Engineering stress-strain diagrams were recorded with a plotter. The reduction in area was calculated by measuring the diameter on the fracture zone of the broken specimens. High temperature tensile tests conducted at different temperatures and strain rates on OFHC copper are summarized in Table 1.

Table 1. Experimental program for high temperature tensile tests conducted at different temperatures and strain rates on OFHC copper. (IGS is initial grain size, T is temperature).

IGS (μm)	T ($^{\circ}\text{C}$)	Cross-head speed (mm/min)					
		1	2	5	10	20	50
25	500		X		X		X
25	700		X		X		X
50	-		X		X		X
50	300		X		X		X
50	405				X		
50	500		X		X		X
50	700	X	X	X	X	X	X
100	-				X		
100	500		X	X	X	X	X
100	700	X	X	X	X	X	X
100	750		X		X		X
150	700		X		X		

Rupture zones of elevated temperature specimens which are pre-investigated by stereo microscope (Leica Mz 125, Heerbrugg, Switzerland) were investigated by

SEM (Jeol Jsm T-330, Tokyo, Japan) at high magnifications. SEM investigations contributed to the determination of fracture type.

3. Results and Discussion

High temperature tensile behavior of copper materials which have different initial grain sizes is shown in Figure 1. The local stress increases observed after necking at the curves 500-2, 700-10, 700-2 and 700-1 in Figure 1 (b) could be caused by the effects of dynamic recrystallization and grain refinement. Strain hardening covering the dislocation generation, multiplication and intersection are significant as the strain increases in the early stages of deformation. The flow stress quickly increases since the dynamic softening caused by cross-

slip is not enough to overcome the effect of strain hardening. Dynamic recrystallization usually takes place due to the buildup of stored energy sufficiently after the critical strain is exceeded. Hence the increasing rate of flow stress decreases rapidly up to the peak value. The softening occurs due to the dislocation climb and dynamic recrystallization beyond the peak flow stress, during which the softening effect dominates: the flow curve decreases continuously until the dynamic balance between strain hardening and dynamic softening is achieved [35].

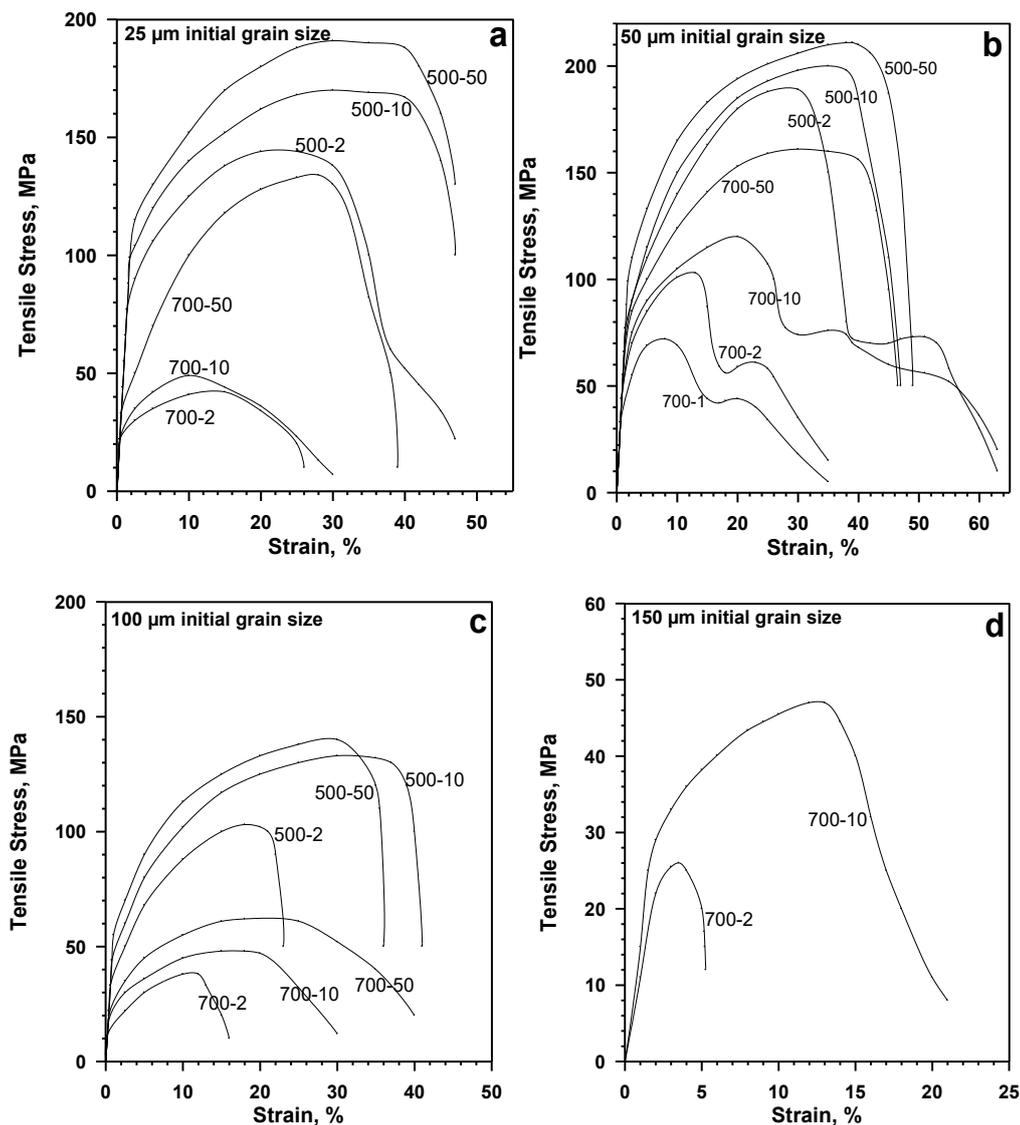


Figure 1. High temperature engineering tensile behavior of copper materials which have different initial grain sizes. Test temperatures ($^{\circ}\text{C}$) and cross-head speeds (mm/min) are given on the curves.

Shear strain is often produced by dislocation slip and/or deformation twinning, especially when a metal or alloy is plastically deformed at low temperatures and strain rates. Grain rotation, grain boundary sliding, and diffusion are other deformation mechanisms, but these

mechanisms only become important at relatively high temperatures, especially when the grain sizes are relatively large [20].

It is reported that one grain layer slides relative to the other and produces a shear stress in the process: plastic deformation has occurred due to the upper layers displaced to the right with respect to the lower layer of the grains [21]. This requires grain boundary sliding and is the basic mechanism in super plasticity.

It is also reported in another study on nanocrystalline materials that the dominant mode of superplasticity is grain-boundary sliding. Van Swygenhoven et al. demonstrated by molecular-dynamics simulations that grain-boundary sliding is the primary deformation mechanism in nanocrystalline materials [36].

Some models about the parameters such as the grain size, grain boundaries, slip bands and dislocation movements have been developed. A widely used model as an explanation for the empirical Hall–Petch relationship relating the yield strength of a polycrystalline material to its grains reports that the pile-up of dislocations against grain boundaries can lead to localized high-intensity stress concentrations, especially in planar slip materials. [37].

3.1. Effect of Grain Size

Initial grain size has a strong influence on the flow stress behavior and the recrystallization kinetics [38]. The flow stress tends to oscillate if $D_0 < 2 D_{rex}$, since there are sufficient grain boundaries for nucleation as suggested by Sakai and Jonas [39].

Grain boundaries are the source and sink of dislocations in a wide range of grain sizes. Particularly in nanocrystalline FCC metals, the emission of partial dislocations from grain boundaries is important. This leads to stacking faults and the formation of deformation twins. When the grain sizes decrease, grain rotation and grain boundary sliding becomes dominant, which can be supported by diffusion and dislocation activities [20].

The effect of grain size on reduction in area and tensile strength at 500 and 700 °C is seen in Figures 2 and 3 respectively. Effect of grain size on reduction in area was investigated in three different regions (Figure 2). Reduction in area is high on the specimens which have initial grain sizes of 25 and 50 μm corresponding the region I. Ductility is not dependent on temperature and strain rate in this region. Ductility decreases with the increasing grain size in region II which is a transition region. Ductility is influenced significantly by the temperature and strain rate in this region. Ductility is low in region III which the grain size increases (150 μm) and is not influenced by the strain rate too much [40].

Strength of the OFHC copper specimens decreases as the initial grain size increases on the deformation at 500 and 700 °C for all strain rates as can be seen from Figure 3. But a peak value at the strengths of the

specimens is seen for initial grain size of 50 μm . This peak value is more obvious for 700 °C than the one for 500 °C especially for lower strain rates.

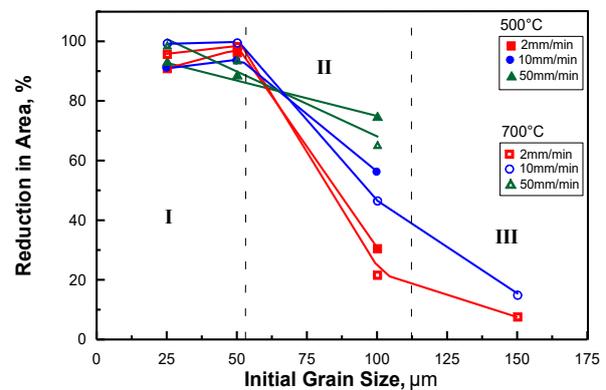


Figure 2. Variation of reduction in area with the initial grain size in the OFHC copper specimens.

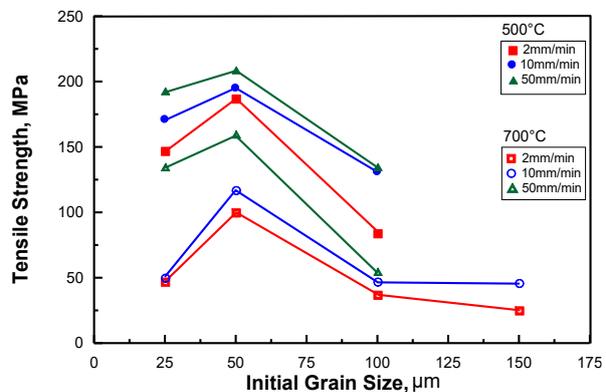


Figure 3. Variation of tensile strength with initial grain size in OFHC copper specimens.

Strength of the OFHC copper specimens decreases as the initial grain size increases on the deformation at 500 and 700 °C for all strain rates as can be seen from Figure 3. But a peak value in the strengths of the specimens is observed for the initial grain size of 50 μm . This peak value is more obvious for 700 °C than the one for 500 °C especially for lower strain rates.

Dynamic recrystallization occurs more easily with increasing temperature [41]. Ductility is high at all strain rates since the dynamic recrystallization ratio is high on the specimens having fine grain sizes at the strain rates at 500 and 700 °C. In the case of large grain sizes, it can be assumed that vacancies formed at the grain boundaries, due to the grain boundary sliding, caused the material to be fractured intergranularly and ductility decreased. Grain boundary sliding occurrence is mentioned in a study on OFHC copper: stress concentrations which can cause cracks and cavities arose in boundary irregularities such as triple points or at grain boundary particles [42]. As the grain size increases, the magnitude of stress concentrations increases.

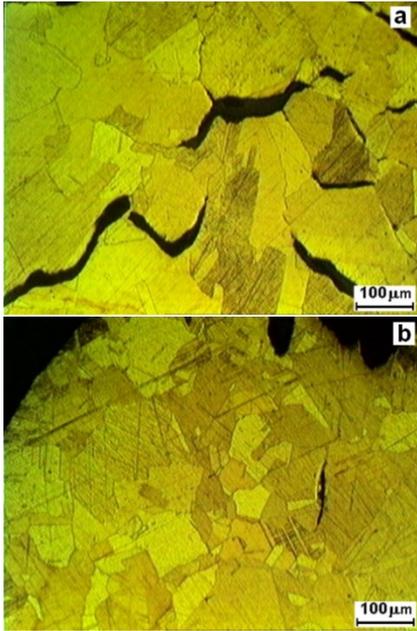


Figure 4. Microstructures of OFHC copper having an initial grain size of 100 μm tested at 750 $^{\circ}\text{C}$ (a) a strain rate of 2 mm/min, (b) a strain rate of 50 mm/min.

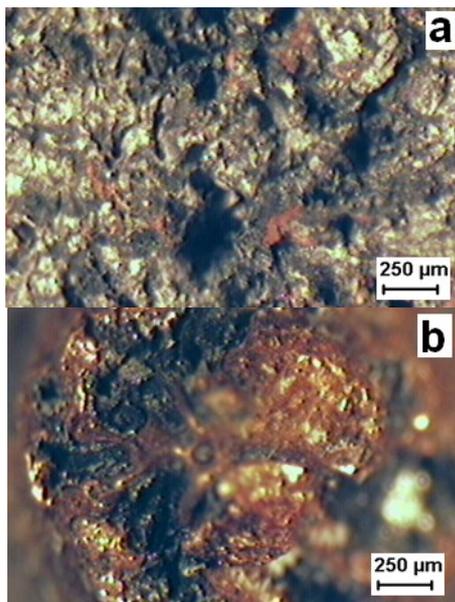


Figure 5. Fracture surfaces of OFHC copper having an initial grain size of 100 μm tested at 750 $^{\circ}\text{C}$ (a) a strain rate of 2 mm/min, (b) a strain rate of 50 mm/min.

Microstructures and fracture surfaces of OFHC copper having an initial grain size of 100 μm tested at 750 $^{\circ}\text{C}$ (a) a strain rate of 2 mm/min, (b) a strain rate of 50 mm/min are seen in Figures 4 and 5 respectively. It is seen that cavities are formed and intergranular fracture occurs due to grain boundary sliding at a strain rate of 2 mm/min (Figure 4 (a) and Figure 5 (a)); the amount of grain boundary sliding slightly decreases as the dynamic recrystallization increases for higher strain rates (Figure

4 (b) and Figure 5 (b)). The intercrystalline brittleness was investigated in the tin bronzes [43] and this brittleness was found to be mainly due to the grain boundary sliding mechanism, which generates a tensile stresses concentration at the grain boundary.

3.2. Effect of Temperature

The effect of temperature on ductility and tensile strength is seen in Figures 6 and 7 respectively. As can be seen from Figure 6, the values of reduction in area of OFHC copper specimens which have initial grain sizes of 50 μm are high and not influenced by the increasing temperature. Ductility is preserved at higher temperatures (500 $^{\circ}\text{C}$ - 0.57 T_m and 700 $^{\circ}\text{C}$ - 0.72 T_m) as a result of the occurrence of dynamic recrystallization. An evaluation of potential mechanisms responsible for low ductility at high temperatures indicates that grain boundary sliding is the dominant deformation mode in CuNiBe alloy at intermediate temperatures due to the high matrix strength associated with the high density of semi-coherent precipitates [22].

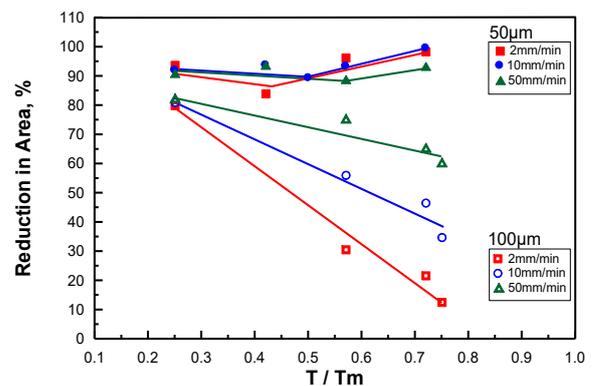


Figure 6. Variation of reduction in area with temperature in OFHC copper specimens.

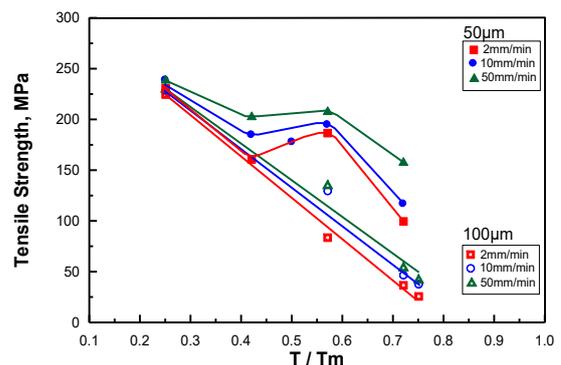


Figure 7. Variation of strength with temperature in OFHC copper specimens.

The reduction in area of specimens which have initial grain sizes of 100 μm decreases quickly by the increase in temperature. Increasing temperature and grain size increase the effect of intergranular fracture by grain boundary sliding. It is understood that the decrease in ductility is very sensitive against the strain rate.

Ductility increases with increasing strain rate. Possible causes are the increase in dynamic recrystallization ratio with the increasing strain rate and more difficult intergranular fracture behavior. Tensile strength decreases when temperature increases as can be seen from Figure 7. Tensile strength of copper specimens decreases with the increasing temperature for initial grain size of 100 μm but there is a slower decrease at the tensile strength of copper specimens having an initial grain size of 50 μm .

3.3. Effect of Strain Rate

The effect of strain rate and temperature has also been investigated in copper: a decreasing strain rate reduces ductility at a constant temperature. An increase in temperature up to 400 or 500 $^{\circ}\text{C}$ also causes a decrease in ductility [42].

The effect of strain rate on reduction in area and strength at 700 $^{\circ}\text{C}$ is shown in Figures 8 and 9 respectively. It is known that decreasing the strain rate decreases the equicohesive temperature and thus increases the tendency of intergranular fracture [44]. Effect of strain rate on reduction in area was studied in three regions for 700 $^{\circ}\text{C}$ and initial grain size of 100 μm by Lim and Lu [27]. In region I, grain boundary sliding and intergranular fracture is dominant in lower strain rates; ductility is low and not dependent on the strain rate. Region II is the transition region; ductility increases with the increasing strain rate by the influence of the dynamic recrystallization. In region III, dynamic recrystallization is the dominant mechanism; ductility is high and not dependent on the strain rate. As can be seen from Figure 6, the ductility values of the specimens having initial grain sizes of 25 and 50 μm are high and not dependent on the strain rate. Ductility increases with the increasing strain rate on the specimens having initial grain sizes of 100 μm . Ductility values of the specimens having initial grain sizes of 150 μm are low and not dependent on the strain rate.

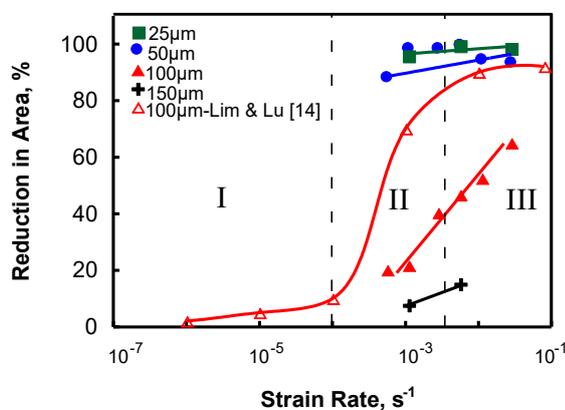


Figure 8. Variation of reduction in area with strain rate at 700 $^{\circ}\text{C}$ in OFHC copper specimens.

The effect of strain rate on tensile deformation was also investigated for the materials except for the copper. The high micro-shear banding frequency seen at low strain rates was found to be associated with an increased incidence of grain boundary sliding for an ultrafine-grained aluminum alloy. An increased slip level is required at higher strain rates since grain boundary sliding is limited. Micro-shear bands produced under these conditions quickly turn into macro-shear bands leading to failure [45].

According to Figure 8, the ductility does not change with the increasing strain rates for the lower initial grain sizes, but the ductility increases as the strain rate increases for the higher initial grain sizes. The slopes of the strength-strain rate curves seen in Figure 9, showing the increasing trend of tensile strength of copper specimens with the increasing strain rate, are consistent with the study of Lim and Lu [27].

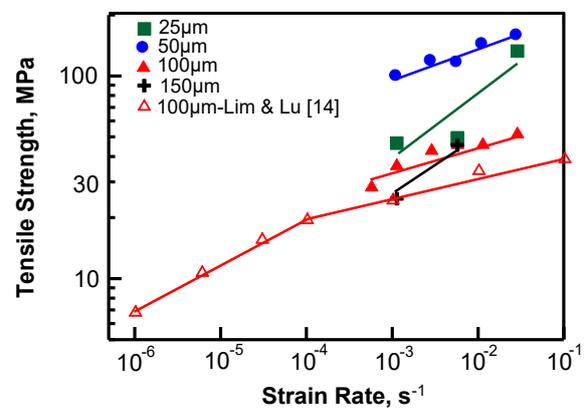


Figure 9. Variation of tensile strength with strain rate at 700 $^{\circ}\text{C}$ in OFHC copper specimens.

Fracture mechanism comes out as a result of hot deformation behavior of material. Ductile fracture occurs at low temperatures in FCC materials. Typical ductile fracture was seen on the specimens in which dynamic recrystallization occurred at the studied temperatures (Figure 10), but intergranular fracture was detected on the coarse grained specimens due to grain boundary sliding (Figure 11). In a 3N copper material, tested at 600 $^{\circ}\text{C}$, intergranular fracture was observed at the fractured surface: a lot of small dimples were observed on the grain boundaries. Many intergranular failures were also observed in the microstructure of this copper. The cavities first nucleate at the grain boundaries and become enlarged and chained together during deformation [41].

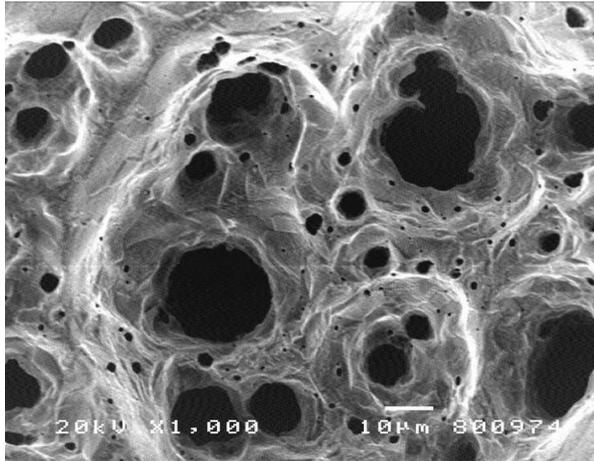


Figure 10. SEM image of a copper specimen which is broken in a ductile manner due to dynamic recrystallization; 700 °C (0.72 T_m), initial grain size: 50 μm , cross-head speed: 10 mm/min.

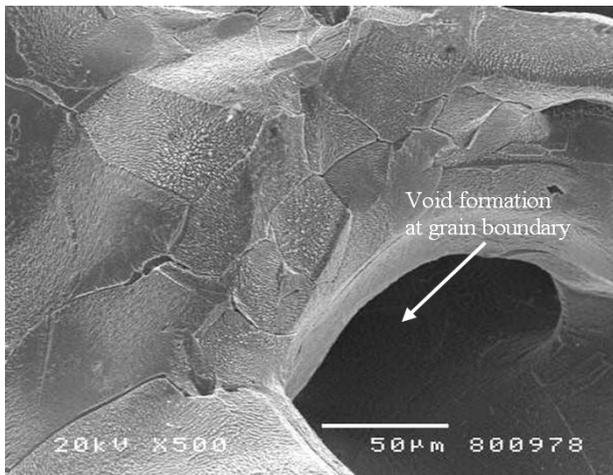


Figure 11. Void formation at the grain boundaries as a result of intergranular fracture at elevated temperatures; fracture surface of tensile specimen; 750 °C (0.75 T_m), initial grain size: 100 μm , cross-head speed: 2 mm/min.

4. Conclusion

Samples of different initial grain sizes were subjected to tensile tests at different temperatures and cross-head speeds to optimize the conditions to achieve maximum ductility in terms of initial grain size, process temperature and deformation rate in OFHC copper.

The optimum initial grain size is 50 μm , from the viewpoint of dynamic recrystallization, which can be responsible for the increase of strength and ductility at the temperatures and strain rates studied. The highest ductility for the initial grain size of 50 μm was obtained at 700 °C and a strain rate of $5.6 \times 10^{-3} \text{ s}^{-1}$ (62.4% strain and 99.6% reduction in area).

Fracture mechanism in OFHC copper is dependent on the parameters such as temperature, strain rate and grain size. Ductile fracture was observed at room temperature and it is the dominant fracture mechanism at elevated temperatures which have the conditions that dynamic recrystallization to occur. Intergranular fracture was observed under the conditions when dynamic recrystallization did not occur.

Working at temperatures and strain rates in which the dynamic recrystallization is the effective mechanism provides the easier production of the parts which are hard to be shaped in the real application conditions. This observed ductility increase for copper may also be used as a model for the other metals especially having face centered cubic (FCC) structure.

Author's Contributions

Selim Yıldırım: Conceptualized the methodology, made investigations, performed the experiments and helped in manuscript preparation.

Mustafa Merih Arkan: Made investigations, wrote and edited the manuscript, interpreted the results.

Ethics

There are no ethical issues after the publication of this manuscript.

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