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Effect of Temperature on Microstructure Evolution of a Nikalin Alloy during Multidirectional Forging

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ABSTRACT

In this study, the microstructural changes of a Nikalin alloy based on Al-Zn-Mg-Ni-Fe-Zr system have been investigated during multidirectional forging (MDF) in the temperature range of 440 to 500 °C and strain rate of 10-3 s-1 with total strain of 2.8 and 6.3. The microstructural images showed that continuous dynamic recrystallization (CDRX) is the main mechanism of recrystallization. Based on the CDRX mechanism, it can be concluded that performing MDF at higher total strain leads to an increase in the number of recrystallized grains and the primary coarse grains are gradually replaced by recrystallized fine grains. Grain refinement was also accelerated by increasing the temperature of MDF, although the grain size increased slightly.

Keywords: Al–Zn–Mg–Ni-Fe-Zr alloy, Nikalin, Multidirectional Forging (MDF), Grain Refinement, Continuous Dynamic Recrystallization

Introduction

Multidirectional forging (MDF) process can be used to achieve a fine-grained structure [1-4]. This processing method does not need a special device and is an efficient method which has a high potential for producing relatively large samples; therefore, this technique can be suitable for industrial applications. Multidirectional forging is basically a type of compression process that is repeated by changing the axis of strain (i.e., x → y → z → x...) applied in each step. Because the workpiece does not change in shape or size, large plastic strains can be applied during high repetitive passes at low-to-high temperatures [5,6]. MDF provides information about the microstructural evolution under different deformation conditions at any temperature and strain rate. As a result, MDF can be a powerful scientific tool for studying structural changes during large strain deformation [5, 6]. Sitdikov et al. [7] studied the effect of isothermal MDF on microstructure development in the Al-Mg alloy with nano-size aluminides of Sc and Zr at the temperatures of 325 °C and 450 °C and at a strain rate of 10-2 s-1 to a total strain of 8.4 with a pass strain of 0.7. Zhao et al. [8] investigated the effect of temperature of MDF on the grain structure at strain rates of 10−3 s−1 and 5 × 10−4 s−1 for the Al-Zn-Mg-Cu alloy. Also, in another study, [9] the evolution of grain structure during isothermal MDF of an Al-Zn-Mg-Cu alloy at 400 °C and 450 °C with strain rates from 10−3 s−1 to 10−5 s−1 was investigated.

The prime aim of this study is investigation of the effect of strain and temperature to achieve a fine-grained structure during MDF. For this purpose, Al-6.2Zn-0.70Mg-1.07Ni-0.75Fe-0.25Zr alloy as a Nikalin Alloy is produced through the casting process, and then the homogenization process is performed on it. Finally, the MDF process is performed on the sample to break the casting microstructure and to reach a fine-grained material. Also, the microstructural changes are investigated under different deformation temperatures of MDF process.

Experimental Methods

The melting of Al-7Zn-2.8Mg-0.7Ni-0.55Fe-0.2Zr alloy was performed in an electric resistance furnace. Nickel, iron, zinc, and magnesium were added into the molten aluminum in the form of pure metals, while zirconium was added as the main binary alloy (Al-15wt% Zr). The melt with a temperature of ~780 °C was cast in a sand die to obtain a cubic ingot with dimensions of 90×80×20 mm3. Then it was quickly cooled in water. The chemical composition of the experimental alloy according to the analysis of the spark emission spectroscopy (SES) is given in Table 1, from which it can be seen that the actual composition is close to the target one. The ingot was homogenized at 450 °C /3 h + 540 °C/3 h. The microstructure of the homogenized Nikalin alloy is in the form of coarse grains with an average size of 109 μm and the phase Al9FeNi precipitated in the grain boundaries of the aluminum solid solution phase (Al), as seen in Fig. 1.

Zn	Mg	Ni	Fe	Zr	Al
7.3	2.3	1.07	0.75	0.25	Bal.

Table 1: Chemical composition of experimental alloy (wt%)

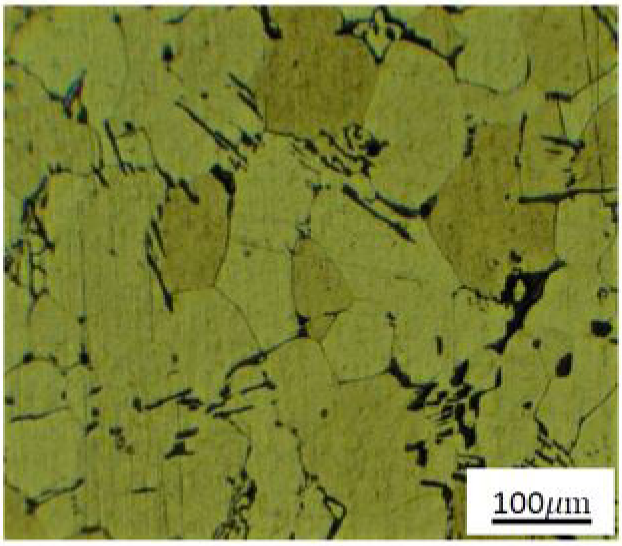


Figure:1 Optical micrograph of the homogenized alloy

For MDF processing, rectangular cube samples with dimensions of 15 × 13.8 × 8.4 mm^3 (with a ratio of 1.8: 1.65: 1) were prepared from the homogenized ingot [6]. The MDF process was performed by the Cronics-25 hot compression test machine, which is equipped with a fully controlled furnace with the computer equipment. In the test design, mica sheet and boron nitride powder were used as lubricants to reduce friction and prevent the jaws from reacting with the sample at high temperatures. After the furnace temperature reached the desired temperature, the sample was kept inside the furnace and between the two jaws for 10 minutes to achieve isothermal condition between the furnace and the sample. Then MDF was performed at a strain rate of 10^{-3} s^{-1} and at different temperatures of 440 °C, 470 °C, and 500 °C. In this processing, each sample underwent plastic deformation up to a total strain of $\Delta\varepsilon\Sigma = 6.3$. For this purpose, the height of the sample was halved in each pass; therefore, the equivalent strain is $\varepsilon = 0.7$ in each pass. Between the passes, the sample was rotated 90˚ according to the cycle z → x → y → z → x. the MDF process was performed up to 9 passes, and the workpiece was quickly quenched in water after each pass, in order to maintain the microstructure. The schematic of the MDF processing is shown in Figure- 2.

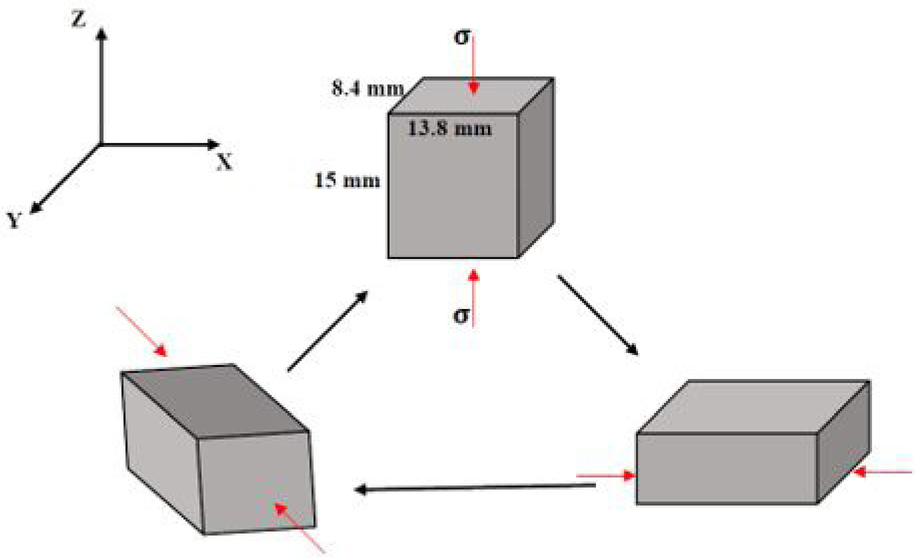


Figure 2: Schematic map of MDF processing with application of stress σ

To study the microstructure, the specimens were first cut parallel to their longitudinal axis, which is in line with the last direction of pressure. The microstructures of the homogeneous and multidirectional forged samples were examined using an Olympus PME3 optical microscope (OM) made in Japan. After the grinding the samples, polishing treatment was performed using aqueous suspension of 0.3 micron alumina powder. These samples were etched in a solution containing 90% H_2O and 10% HF at room temperature.

Results and Discussion

Fig. 3 shows a OM image of the microstructure of the sample of Nikalin alloy forged at the temperature of 440 °C and a strain rate of 10^{-3} s^{-1} with $\Sigma\Delta\varepsilon = 2.8$. As can be seen, the particles of the Al_9FeNi eutectic phase are elongated and are finer than those of the homogenized state. In addition, a small number of grains are subdivided into new fine grains. These new fine grains often develop inside the grain. The recrystallized grains are continuously formed by increasing the angle of the sub-boundaries by increasing the strain to 2.8. Also, the number of recrystallization grains formed in the interior of the grain gradually increases with increasing the total strain. Based on the previous research, the dominant mechanism for grain refining is continuous dynamic recrystallization (CDRX) [8].

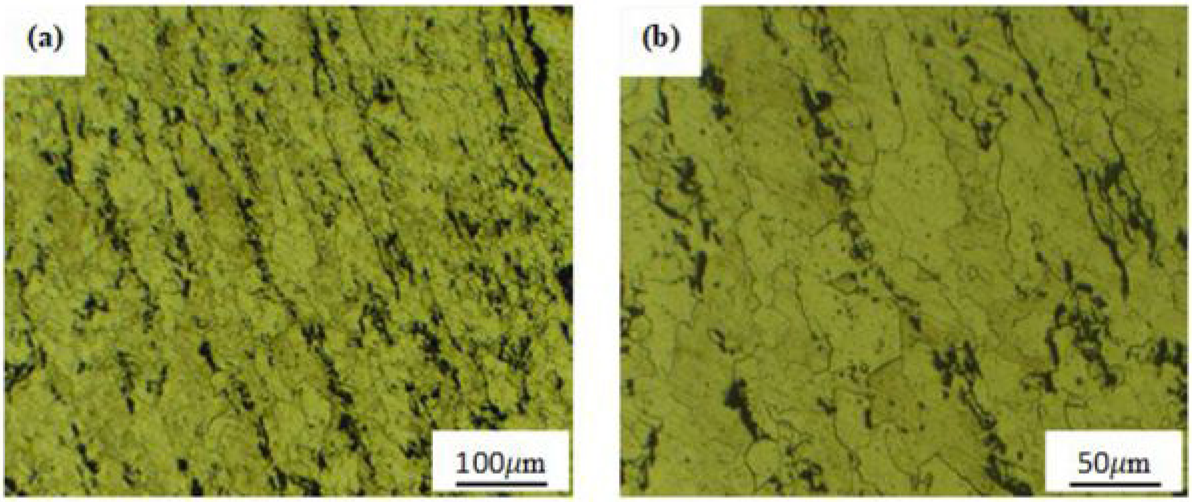


Figure 3: Optical microscopic images of the multidirectional forged sample processed at 440 °C and 10^{-3} s^{-1} with $\Sigma\Delta\varepsilon = 2.8$ in two magnifications

By increasing the total strain to $\Sigma\Delta\varepsilon = 6.3$ as shown in Fig. 4, it can be seen that a fine-grained structure with an average grain size of about 12 μm has been developed in almost the entire volume, while there are still some large coarse grains. By applying more strain in the continues passes and changing the sample direction in each pass, the deformation bands expand and intersect in different directions throughout the sample, followed by conversion of the low angle grain boundaries (LAGBs) to high angle grain boundaries (HAGBs) by the CDRX mechanism. As a result, sub-grains in non-recrystallized regions are replaced by fine-grained recrystallized grains [10, 8]. This microstructure resulting from MDF is characterized by a two-dimensional grain size distribution, as shown in Fig. 4 (c). The microstructure has small grains with an average size of about 8 μm and large grains with an average size of about 17 μm. Therefore, the average value of 12 μm is obtained for the grain size.

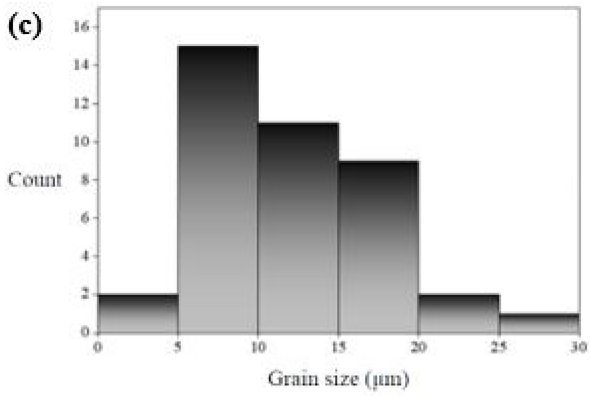


Figure 4: (a, b) Optical micrographs of the MDFed sample forged at 440 °C and 10^{-3} s^{-1} with $\Sigma\Delta\varepsilon = 6.3$ in two magnifications (c) Grain size distribution

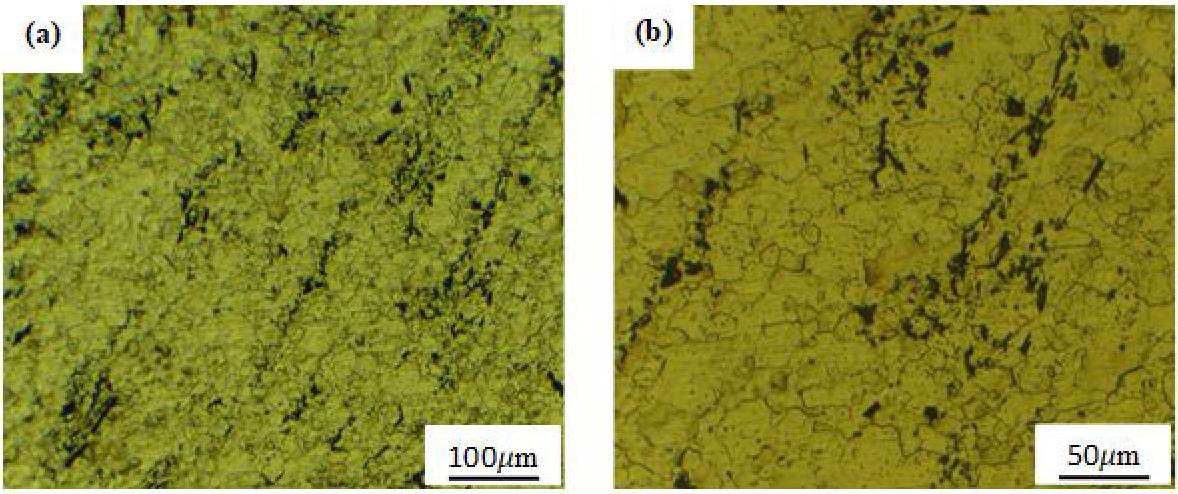
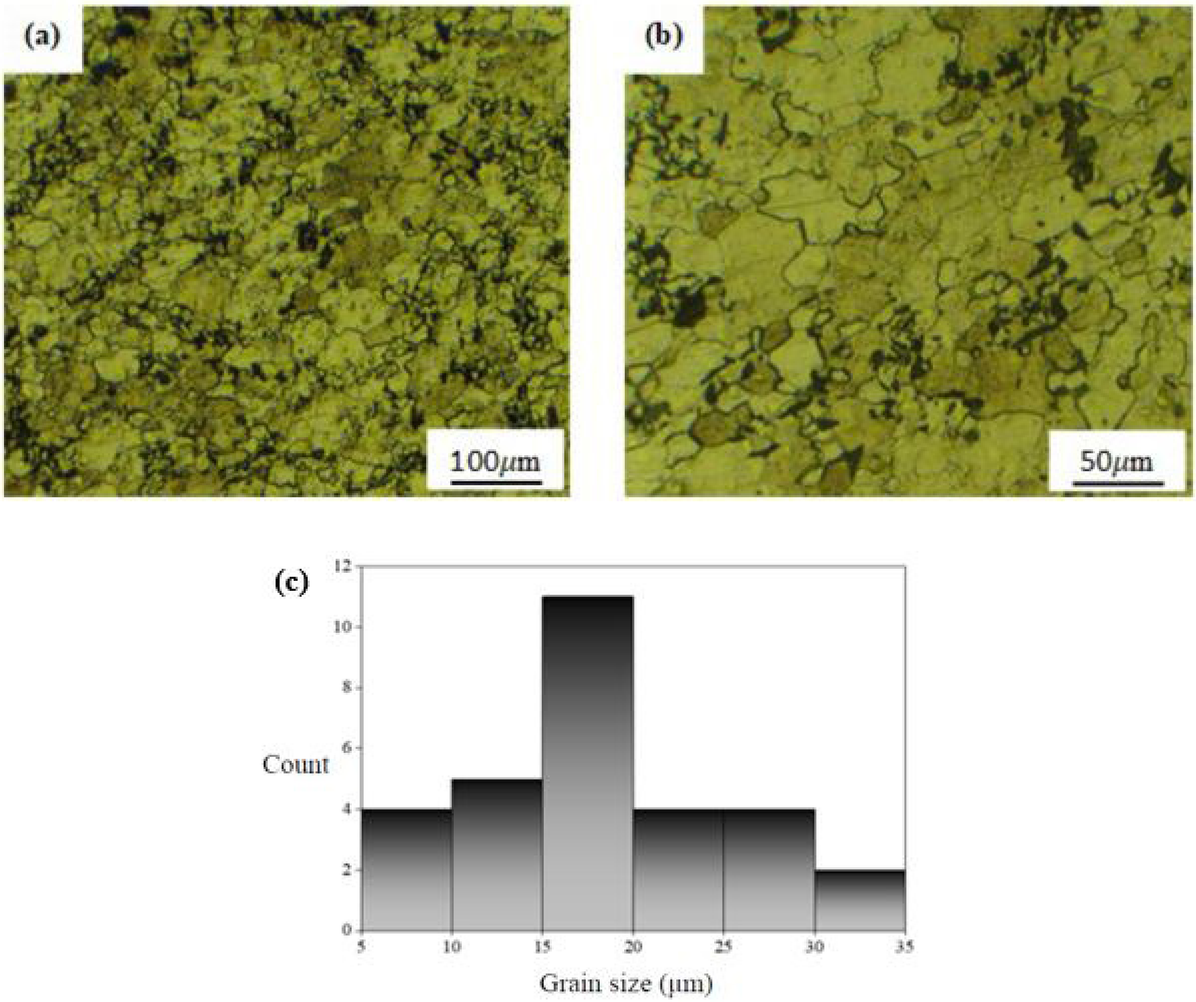


Figure 5: shows a OM image of the microstructure the multidirectional forged sample processed at temperature of 470 °C and a strain rate of 10^{-3} s^{-1} with $\Sigma\Delta\varepsilon = 6.3$

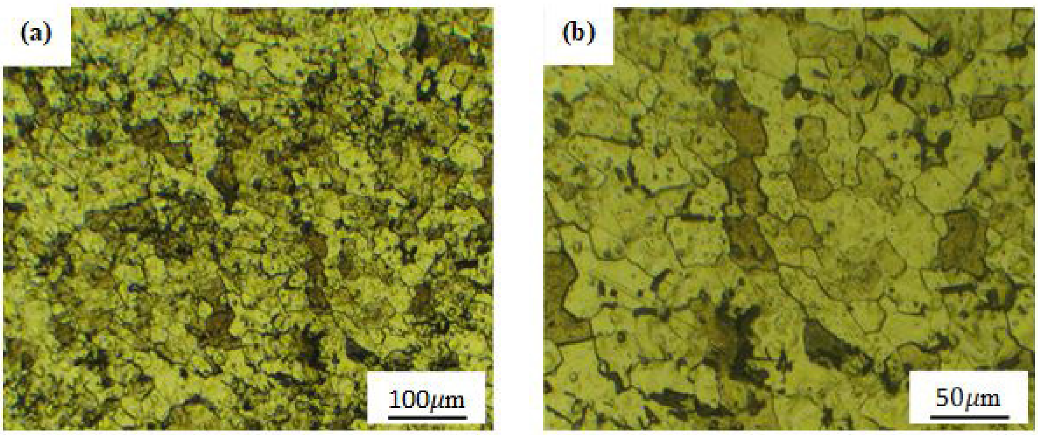
Due to the microstructure, the eutectic phase of Al_9FeNi is crushed and distributed evenly throughout the structure. Comparing Fig. 5 with Fig. 4, it can be said that the microstructure has become slightly larger with increasing temperature due to the growth phenomenon which is activated by temperature and time. As a result, with increasing the temperature, the mobility of grain boundaries increases; as a result grain size is coarser in comparison against the lower temperatures as shown in Fig. 5 (c) [11]. In addition, with increasing the MDF temperature, the grain refinement effect is faster and more volume of the fine-grained material is obtained. The formation of the main grain boundaries is accelerated by increasing the temperature of MDF, as a result more recrystallized fine-grained with the same total strain are produced at higher temperatures [8].



(27)

Figure 5: (a, b) Optical microstructure of MDF sample processed at 470 °C and10^{-3} s^{-1} with ΣΔε = 6.3 in two magnifications (c) Grain size distribution

As shown in Fig. 6, the microstructure of the forged sample at 500 °C becomes larger in comparison with the samples processed at 440 °C and 470 °C. According to Fig 6 (c), this fact is confirmed by the measured average grain size of 22 μm. As can be seen in Figs. 4, 5, and 6, the MDF process continuously refines the grain structure by imposing the total strain of ΣΔε = 6.3. When CDRX is the main recrystallization mechanism, the number of recrystallized grains gradually increases with increasing total strain, and the primary coarse grains are gradually replaced by fine recrystallized grains. In addition, with increasing the temperature of MDF, the grain refining effect was accelerated and the grain size increased slightly.



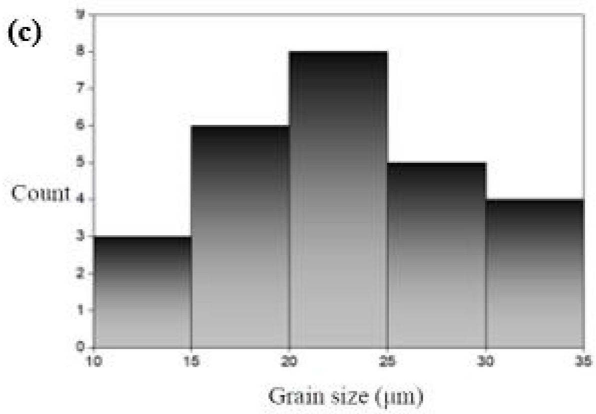


Figure 6: (a, b) Optical micrographs of the MDF sample processed at 500 °C and 10^{-3} s^{-1} with ΣΔε = 6.3 in two magnifications (c) Grain size distribution

Conclusions

As the total strain increased from ΣΔε = 2.8 to ΣΔε = 6.3, the recrystallized grains increased so that the primary coarse grains were gradually replaced by fine recrystallized grains, while some coarse grains remained.

As the temperature increases, the microstructure becomes slightly coarser. In addition, with increasing the MDF temperature, the grain refinement effect is faster and more volume of fine-grained material is obtained.

Performing the MDF processing on Nikalin alloy leads to a fine-grained microstructure and grain size becomes less than 20 µm. Also the phase of Al_9FeNi become finer and its distribution is uniform throughout the microstructure.

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