Muhammed Fatih Kilicaslan^{a*}, Saad Ibrahim Elburni^b, Bekir Akgul^c

^a*Department of Fundamental Sciences and Engineering, Sivas University of Science and Technology, 58000, Sivas Turkey*

^b*Department of Materials Science and Engineering, Kastamonu University, 37000, Kastamonu, Turkey*

^c*Department of Aeronautical Engineering, Sivas University of Science and Technology, 58000, Sivas, Turkey*

*fatihkilicaslan@sivas.edu.tr

THE EFFECTS OF Nb ADDITION ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF MELT SPUN Al-7075 ALLOY

ABSTRACT

The mechanical properties of the Al-7075 material, which is used in important areas such as automotive, aviation and defense industry, are still being studied by researchers. In this study, the effects of different proportions of Nb additives on the microstructure and mechanical properties of the rapidly solidified Al-7075 alloy was investigated. Rapid solidification processes were carried out with a single roller melt spinner at a disk surface speed of 25 m / s. Microstructure characterization was performed by using a scanning electron microscopy (SEM) and X-ray diffraction. According to the results, the Nb additive significantly increased the micro hardness of the Al-7075 alloy. The microhardness of the sample added with 0.5% by weight of Nb is 0.9 GPa. This value is 3 times higher than the sample without Nb added. The Nb contribution has led to modification of the dimensions and shapes of both α -Al and intermetallic phases. Nb addition reduced the average grain size from 9.1 μm to 2.46 μm .

Keywords: Metals and alloys; microstructure; metallurgy

INTRODUCTION

Aluminum and its alloys, especially Al-7075 series ones, are frequently used in aviation and automotive industries due to their low density, high strength, ductility and fatigue resistance [1–4]. Although the Al-7075 alloy has excellent properties, researchers are still working to improve some of the properties of this material [2,5,6]. In order to further improve the mechanical properties of aluminum alloys, researchers make microstructure modifications by means of various additive elements such as Co, Ni, V, Sc, Ti, B and / or use new generation production techniques [7–9]. Methods such as precipitation hardening [10–12], deformation hardening [13,14], semi-solid casting [15,16] and/or various rapid solidification[2,5,17–19] are applied to aluminum alloys whose strength cannot be increased by quenching. Many alloying elements are added to the aluminum alloy to refine the grain size by operating the heterogeneous nucleation mechanism. Nucleation centers are increased

by the formation of high melting intermetallic phases such as MgZn_2 , Al_3Sc , Al_3Nb , Al_2CuMg , $\text{Al}_7\text{Cu}_2\text{Fe}$, MgZn_2 , Al_6AgMg_4 . In this way, the grain size gets thinner as the number of grains in the structure increases [9,12,14,20]. Additionally, melt spinning, a kind of rapid solidification techniques, is a new generation production method that allows the production of superior aluminum alloys. The melt spinning method stands out because it produces high cooling rates such as 10^7 K/s and is suitable for mass production [6,9,21,22]. Rapid solidification process in aluminum alloys provides benefits such as reduction of segregation, grain size refinement, suppression or elimination of coarse segregation phases, modification of secondary phases in terms of size and shape, increase of chemical homogeneity, minimization of time or temperature in aging processes [2,5,21,23,24]. Unlike traditional production methods, the grain size of the materials produced by rapid solidification is finer. As is known, the mechanical properties of fine-grained structures such as hardness, tensile strength, and fatigue strength are better than coarse-grained structures [2,5,6,9,22,25,26]. In traditional production methods, an alloying element such as Ti-B can be added as a grain refiner [27]. It is also known that element Nb is added to various alloys as a grain refiner and strength enhancer [28,29]. In Wang's study, it is stated that Nb and Al elements form Al_3Nb intermetallic compound and this phase is located approximately in the center of the grains and plays a role in initiating nucleation [28]. The Nb element reduces structural undercooling by operating the heterogeneous nucleation mechanism to form co-dispersed fine particles in the structure [26,29]. Nowak et al. emphasized in their study that Nb-B addition is more effective than commercial Ti-B addition in grain refinement [29]. In this way, a microstructure with homogeneous distribution and minimum segregation is obtained within the structure. The melt spinning method also minimizes the chemical composition segregation seen in conventional methods [26]. According to the study conducted by Liu et al. in 2021, grain size and micro segregation decrease with increasing cooling rate [26]. Unfortunately, it is known that there are micro-chemical composition differences between the fast-solidifying region and the slow-solidifying region in traditional production methods. Therefore, in the melt spinning method, since the whole structure solidifies rapidly, there is no difference in composition distribution. However, to our best knowledge, there is not enough information in the open literature on the effects of Nb doping on the microstructure and mechanical properties of rapidly solidified Al-7075 alloys. In addition, T6 heat treatment is applied to increase the strength of Al-7075 alloys. However, as this increases the grain size, it can increase the hardness up to a certain point [30]. For this reason, in the presented study, effect of Nb contribution on microstructure and mechanical properties of Al-7075 alloy produced by melt spinning method is examined. In this way, the effect of Nb addition to Al-7075 alloy known as aviation material and melt spinning method on microstructure and mechanical properties has been demonstrated. Unlike traditional production methods, it has been shown that the hardness can be carried to higher points by forming a very fine grain structure with the melt spinning method. As a matter of fact, the hardness has increased approximately three times without heat treatment.

EXPERIMENTAL

A commercial purity Al-7075 and Nb alloys were used to prepare Al-7075-XNb alloys (X= wt% 0.2 and 0.5) with nominal composition. Al10Nb master alloy with a size of 0.5-3 mm is used as Nb raw material. Samples with 500 g of composition were melted twice for homogeneous mixture in high frequency induction furnace. All sample mixtures were taken

up to 750 ° C and kept for 15 minutes and then poured into an ingot mold at 700 ° C. In addition, the selection of the rotation speed of the copper disc is one of the important parameters affecting the cooling rate and ribbon thickness. According to Qing et al., a cooling rate of 2×10^7 K / s was obtained at 30 m / s disk speed and mechanical properties such as hardness increased with the refining of the grain size [22]. Therefore, the melt spinning processes were performed using a copper wheel with 25 m/s disc velocity with pressurized argon of 400 mbar. As can be seen in Figure 1, cylindrical samples weighing approximately 500 g and 10 cm in length were melted in the high-frequency induction furnace again in the melt spinning process and blown through a nozzle at a distance of 1 mm onto the copper disc with an argon pressure of 400 mbar. As a result, ribbons of approximately 10 mm width and 30 μ m thickness were obtained.

Fig. 1. Melt-spinning process

While melt-spun ribbons were denoted as AA00 (Nb free alloy), AA02 (0.2 Nb added) and AA05 (0.5 Niobium added), as-cast alloys were denoted as CA00 (Nb free alloy), CA02 (0.2 Nb added) and CA05 (0.5 Nb added). All of the specimens fabricated are given in Table 1. The cross-sections of ribbons were selected for the indentation and scanning electron microscopy measurements. The microstructure and phase identification of the samples were investigated by FEI Quanta FEG 250 scanning electron microscopy (SEM), SEM-Energy dispersive spectrometer (EDS) and a Bruker D8 Advance X-ray diffractometer. For microhardness hardness measurements, a microhardness tester with a Vickers tip (HMV-G21D) was used. All tests were performed under the same operating conditions. For all samples, the maximum load was 0.55N.

Table 1. Chemical composition of melt spun Al-7075 alloys (wt. %)

Sample	Si	Cu	Mn	Mg	Cr	Zn	Ti	Nb	Fe	Pb	Sn	Al
AA00/ CA00	0.727	1.64	0.053	2.48	0.263	6.52	0.095	-	0.115	0.009	0.008	bal.
AA02/ CA02	0.727	1.64	0.053	2.48	0.263	6.52	0.095	0.2	0.115	0.009	0.008	bal.
AA05/ CA05	0.727	1.64	0.053	2.48	0.263	6.52	0.095	0.5	0.115	0.009	0.008	bal.

RESULTS AND DISCUSSION

Figure 2 shows how the microstructure is evolved with the addition of Nb in as-cast (Figure 2 (a, b and c)). Accordingly, in the sample without additives (AA00) seen Figure 2 (a), the α -Al grains vary between about 7 μm and 17 μm and their average size is 9.1 μm . In Figure 2 (b), that is, in the sample with 0.2 Nb doping (AA02), the sizes of α -Al grains vary between 1.9 μm and 7.7 μm . Here, the average grain size of α -Al is around 4.5 μm . As for the sample AA05 with 0.5Nb (Figure 2 (c)), the particle size of the α -Al phase varies between 1 μm and 5.6 μm and the average particle size of them is approximately 2.46 μm . Hence, the Nb contribution to the Al-7075 alloy gradually caused a reduction in the particle sizes of the α -Al phases. In addition, with the Nb additive, both the distribution of the α -Al phases became more homogeneous and their shapes transformed from an irregular structure (Figure 2 (a)) to a coaxial structure (Figure 2 (c)). Two factors can be mentioned that make the grains fine and homogeneous. First; As Wang stated in his work, the element Nb increased the heterogeneous nucleation in aluminum and increased the number of grains [28]. In this way, structural undercooling has decreased. According to Nowak, more homogeneous and finer-grained structures are formed due to the addition of Nb [29]. Therefore, it is seen that the data we obtained in our study are compatible with the studies of Wang and Nowak [28,29]. The second factor is that the grains formed during cooling due to the rapid cooling caused by the melt spinning cannot find the opportunity to grow. In this way, it is ensured that almost nano-sized particles are formed. In the study carried out by Meng in 2020, a grain size of approximately 2-4 μm was obtained [25]. In our study, close grain sizes were obtained in the sample with 0.5% Nb added. In this study, the lowest average particle size of 2.46 μm was obtained with a cooling speed of 25 m/s. However, Lin et al. Obtained an average particle size of 1.6 μm at a cooling rate of 40 m/s [2]. This shows that as the cooling rate increases, the grain size decreases.

Figure 2 (d) shows XRD patterns taken from the samples. According to XRD analysis, only well-defined Al crystal peaks are seen in all three samples, and the clear peak of any intermetallic is not visible. Possible reasons for not finding any intermetallics in this study may be that intermetallics have very small dimensions due to rapid solidification and / or their ratio within the microstructure is too low. Because XRD is insufficient in detecting phases less than a certain ratio within the microstructure [31]. On the other hand, in Figure 2 (d), within the small frame indicated by an arrow, the 2θ angles at which Al (111) peaks are formed are seen. As can be seen from the figure, with the Nb doping, Al peaks shift to the right, that is, they occur at larger 2θ values. In the literature, this situation is associated with the dissolution of some of the additive element in Al matrix. On the other hand, in ref. [32], it is stated that in the rapidly solidified Al –Si alloys alloy, transition metals can be dissolved in the Al matrix. Thus, we think that in the samples with the Nb doped, some Nb dissolved in the Al matrix and formed a solute solution. In addition, although the Nb peaks are very uncertain, Wang et al. Stated in 2014 that there is an Al_3Nb phase in the structure [28]. Nb peaks in Wang's study are slightly more pronounced than ours. However, in Wang's work, there is no alloying element other than Nb in Al. Therefore, it is possible to see and distinguish Nb peaks. Considering that there are more than one alloying element and phase in our study, it can be understood that Nb peaks are uncertain. However, it is understood from the SEM and EDS results shown in Figure 3 that the Nb element is mixed into the structure. As a result of EDS number 1, which is thought to be a nano-sized intermetallic phase, the Nb ratio has increased to 5.6%. This result shows that there may be Al_3Nb intermetallic phase emphasized by Wang in microstructure [28].

Fig. 2. Optical micrographs as-cast and XRD pattern taken from melt spun Al7075 alloys showing changes in microstructures with addition of Nb; (a) without Nb; (b) with 0.2 pct. Nb; (c) with 0.5 pct. Nb; (d) XRD pattern of samples AA00, AA02, and AA05

Fig. 3. SEM-EDS analyses from melt-spun AA02 alloy

The SEM micrographs given in Figure 4 shows how the microstructure of the Al-7075 alloy is evolved with the Nb addition. In Figure 4 (a), sharp angular particle-like shaped secondary phase grains are observed in the microstructure of the Nb free sample (AA00). The sizes of these secondary phase particles vary between 0.5 μm and 3 μm , and the average particle size of these secondary phases is around 1.6 μm . In Figure 4 (b), with the 0.2 Nb (sample AA02), the shapes of the secondary phases embedded in the Al matrix are transformed into both particle-like and rectangular block-like structures. In the AA02 sample, the dimensions of the secondary phases varied between 0.2 μm and 2.2 μm , and the average dimensions decreased by 1 μm compared to the Nb sample. When the Nb contribution is increased to 0.5% (Figure 4 (c)), the coaxial rounded / spherical shaped secondary phase particles embedded in the Al matrix draw attention in the microstructure of the AA05 sample. The sizes of the rounded / spherical shaped secondary phases vary between 0.3 μm and 1.7 μm and the average particle size is around 1 μm . The secondary phase turns into spherical by adding 0.5% Nb. Liu stated that by adding Ce at the same rate as we do, the secondary phases become spherical [22]. Therefore, it can be said that Nb and Ce elements operate the same heterogeneous nucleation mechanism. Liu showed in his study in 2018 that the cooling rate also affected the formation of spherical secondary phases. According to Liu, after 30 m/s cooling rate, it turns into a spherical excess fibrous structure [22].

Fig. 4. SEM micrographs of melt spun Al-7075 alloys showing changes in microstructures with addition of Nb of a) without Nb, b) 0.2 and c) 0.5 pct

These kinds of small equiaxed secondary particles are in favor of a structure, generally bring about higher mechanical properties [33]. The occurring of rounded / spherical like secondary phases can be clarified according to the literature as following, because of growth of a seed crystal diffusions controlled, and the radial growth rates of an isolated crystal floating in the metallic melt are essentially isotropic. In addition, solute gradients in the small volume of melt covered by a tiny seed are radial symmetrical, which is also in favor of a rounded outline. In the literature, It has been stated that a higher undercooling level can help these tiny seed crystals, to grow isotropically, resulting in a rounded / globular shape[34]. Moreover, it has been stated that in Al alloys, an increased concentration of modifier agent in liquid phase lead to an increased constitutional undercooling [35]. On the other hand, in reference [19], it has been stated that Nb can form a solid solution in Al-Si-Fe-Nb alloy, as well as diffuse into secondary phases (intermetallics). Hence, in this study we conclude that some Nb atoms played a role as a modifier agent in the liquid Al matrix and led to an increment in the constitutional undercooling, which resulted in the formation of rounded / globular-like intermetallic phases.

In Figure 5 changes of microhardness values with the contribution of Nb are given. The variations in the microhardness values of the specimens with the supplement of Nb agree with the above results, and values and changes of them are given in Figure 5, respectively. Accordingly, while the microhardness value of the base sample (AA00) was 0.31 GPa, it was 0.45 GPa for the 0.2 Nb doped sample (AA02), and 0.92 GPa for the 0.5Nb doped sample (AA05). It means that an increase of approximately 3 times has occurred. It is stated in the literature that modification and refining of the secondary phases in microstructure diminish the probability of crack formation and improve the mechanical properties [36]. Therefore, particle size strengthening caused by the addition of Nb, which brought about refining of coarse secondary phases (Figure 4), can be one possible reason for the increment in the microhardness values. Moreover, as stated above, we concluded that Nb can be dissolved in the rapidly solidified aluminum matrix by looking at the XRD analysis (Figure 2(d)), which is in accordance with the reference [19].

Fig. 5. Changes in the dynamic microhardness values of melt spun Al-7075 alloys with different amount of Nb addition

Hence, other possible cause for the increment in the microhardness values can be solid solution hardening. In addition, Nb atoms can cause distortions in the lattice structure by

acting as the substitutional atom in the structure. Of course, the increase in the density of dislocation also means that this increased the hardness. Lin et al. Stated in their study that the cooling rate was also effective in increasing the density of dislocation. It was determined by Lin et al. that dislocation strengthening had an effect at high cooling rates [2]. The data we obtained in our study supports Lin's approach.

CONCLUSIONS

In this study, it's reported the effects of the addition of Nb on the microstructural properties of melt spun Al-7075 alloys. The results can be summarized as follows:

- Nb addition to Al-7075 alloy gradually caused reduction in particle size of α -Al phases. And with the Nb additive, both the distributions of α -Al phases became more homogeneous and their shapes transformed from an irregular structure to a coaxial structure.
- The Nb contribution to the Al-7075 alloy caused the secondary phases in the matrix to decrease in size and to evolve their shape from a sharp-edged particle-like structure to a coaxial rounded / spherical structure.
- Nb additive made to Al-7075 alloy has caused a significant increase in microhardness values. Approximately 3 times higher microhardness value was obtained in the sample with 0.5% Nb by weight.

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