

The effect of silicon on the homogenization of CDC cast aluminium alloys

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Abstract. The homogenization processes of commercial and modified Al 3003 alloys were investigated. The concentration of Si, Zn and the Mn were varied within the standard specification. Not only the temperature but the holding time of homogenization was also varied. The effects of alloying element on the precipitation process during the homogenization were investigated by optical and scanning electron microscopy. The correlative microscopy was also a very useful method to follow the evaluation of different precipitate. Hot and cold compression tests were used to detect the effect of different precipitation on the formability. It was experimentally showed that just a slight difference in the alloying elements resulted in significantly different microstructure and formability after the same homogenization process.

1. Introduction

Aluminum alloys have been widely used in the aerospace, automotive, and construction industries due to their high specific strength, low cost, and good corrosion resistance [1, 2]. The 3003 aluminum alloy is widely used in the container, packaging, and automobile industry, because of its excellent specific strength, corrosion resistance and formability. The alloy 3003 is typically Al-Mn alloys, but some further dissolved elements also used to improve strength and formability [1, 3, 4]. Such elements are, for example, silicon, iron, copper, magnesium, zinc or even zirconium. The effect of the alloying elements for the precipitations and the mechanical properties of the material are already known [5, 6]. For example, the effect of zinc, silicon and titanium elements for the microstructure and mechanical properties is well known [1, 7]. Small amount of zirconium addition helps to increase strength and provides grain coarsening. The addition of iron, silicon, copper, zirconium and titanium to the alloy has a strong influence on the formation of the precipitates. It is well known that iron and silicon reduce the solubility of manganese in the solid solution and accelerate the rate of precipitation of intermetallic compounds [2, 5, 6]. Fe promotes the precipitation of $Al_6(Mn,Fe)$ while Si promotes the precipitation of α phase $Al_{15}(Mn,Fe)_3Si_2$. Different equilibrium processes occur in the material during heat treatment after casting. In this regard the most important is the so-called δ to α transformation where $Al_6(Fe,Mn)$ transform to $Al_{15}(FeMn)_3Si_2$ [8-10]. In addition, further precipitations will be generated from the aluminium matrix during homogenization [11-13]. The type size and distribution of precipitation formed during homogenization have strong effect on the deformation behaviour, recrystallization and mechanical properties of alloys [14,15]. So the homogenization process is crucial to the optimization of the chemical composition and mechanical properties of the alloy. Controlling the state of precipitation



is very important [16, 17]. In the present study the result of two different homogenization processes were compared for the commercial and the modified 3003. In case the modified alloy the concentration of Si, and Zn and the Mn were increased. Not only the temperature but the holding time was also varied.

2. Experimental

Table 1 present the compositions of the investigated ingots were CDC, (Closed Direct Chill) casted in the industrial environment. Comparing the two alloys the differences in Si, Zn and Cu contents are obvious. Sectioning was used to take samples from the cross section of the ingot at the $\frac{1}{4}$ of the width. The samples were homogenized under two conditions: condition A 510°C 1h, 5h or 10 h, and condition B 540°C 4h step 510°C 1h, 5h or 10h. The heat treatments were performed in laboratory environment. After the homogenization subsequent water quenching was applied. The as casted and the homogenized microstructure were observed by OM and TEM. Microstructure studies were performed using a Zeiss Imager M1m optical microscope. Barker etchant and Zeiss Axiovert M40 MAT optical microscope was used to visualize the grain size with polarized light filter. Further investigations were performed using a Zeiss EVO MA10 scanning electron microscope equipped with EDX. Correlative microscopy was used for direct monitoring the solid phase reaction during homogenization by the Zeiss EVO MA10 scanning electron microscope. The same part of the sample was investigated before and after the heat treatment. Specific sample holder provided the coordinates for the orientation on the sample. The as casted sample were prepared for the OM investigations and the quality of the surface was good enough after the heat treatment as well. The homogenization condition B was used for correlative sample.

For modeling hot or cold rolling, hot and cold compression tests were carried out on the cylindrical samples. The tests were performed by Instron 5982 material testing equipment. The cold and hot tests were carried out at 23 ° C and 400 ° C respectively. 1 / sec deformation rate with a 60% reduction were applied in both cases. The heating and holding time of pressing pieces was provided by an inductor. After the cold compression test, a subsequent heat treatment was carried out in 500 ° C in a furnace for 1 hour and then the pieces were cooled into water to control the driving force for the recrystallization. After the examinations the data were evaluated, namely comparison of true and engineering stresses and deformations, true stress and true deformation diagram, and the deformation strength chart. The microstructure was also investigated.

Table 1. Chemical composition of the specimens, wt%

identification	Si	Mg	Mn	Fe	Zn	Cu	Cr	Ti	Al
XH	0,79	0,01	1,61	0,25	1,49	0,04	0,002	0,01	bal.
X8	0,06	0,006	1,09	0,2	0,02	0,52	0,001	0,125	bal.

3. Results

The Figure 2 shows the initial as casted microstructure of the samples, no significant difference can be observed. Only the grain size is smaller in case of X8 sample. Typical coarse Al₆(Fe, Mn) particles were formed during casting at the grain boundary of dendrites. Figure 2-3 present the LM image of the samples after the homogenization under 6 conditions. After performing the homogenizing heat treatments at lower temperatures, optical microscopic examinations suggest that the formation of the second phases is started after one hours in both alloys, which are increasing with time. The difference is observed in the distribution of the precipitations, as the second phase in the high silicon alloy grows around the eutectic, while the low silicon-containing alloys tend to appear along the grain boundary. Very fine precipitations can be observed in the matrix but precipitation free zones around the primary particles still exists. The effect of the temperature and time of the homogenization is different for the two different composition. Increasing the time and the temperature the number of fine particles was also increased. The dissolution of the primary particles also can be observed after 10 hours treatment of X8 alloy. The SDAS value seems to be also smaller which cannot be the result of the homogenization but the inhomogeneity of the initial casted sample, which was different for each case. So, correlative

investigations were performed to clarify this effect. The Figure 4-5 show the result after the heat treatment condition B. It is obvious that the primary particles were nearly unaffected in both alloy even after 10 hours treatment, just a slightly coarsening was observable. The existing of the precipitation free zone is not convincing. But zones with different type of particles is very clear especially at the alloy with lower Silicon concentration. The distribution of zones with very fine precipitates with size less than $1\ \mu\text{m}$ associated with primary dendrites morphology. So it is clear that those formed from the supersaturated solid solution dendrites not from the dissolution of the primary $\text{Al}_6(\text{Fe}, \text{Mn})$. Other zones with coarse precipitates and also needle like precipitates are located between the dendrites. The volume fraction of this zona is much higher after 10 hours. EDX analysis confirmed that the fine precipitates are Al-Mn while the needle like is Al-Fe rich. Because of the small size of the particle other methods with higher resolution are needed for more precise identification. These kinds of needles may have a harmful effect on the mechanical properties especially of the alloy because these sharp boundaries may be stress concentrators. The formability also depends on the morphology and distribution of precipitates. The hot press curves and the microstructures were find identical after the different treatments. But the cold curves behave quite different. The Figure 6 introduces the hardening curve after cold compression tests of the two alloys were homogenized under condition A. The Figure 7 shows the microstructure on the cross section parallel to the axis of the cylindrical sample after cold compression test and annealing heat treatment. The hardening curves of the samples were homogenized different time are similar in case of X8 alloy. Only a small part of the recrystallized volume was detected. Alloy XH where the concentration of alloying elements (Si, Mn, Zn) is higher but the precipitation was finer behaving a very different hardening, especially after 10 hours homogenization.

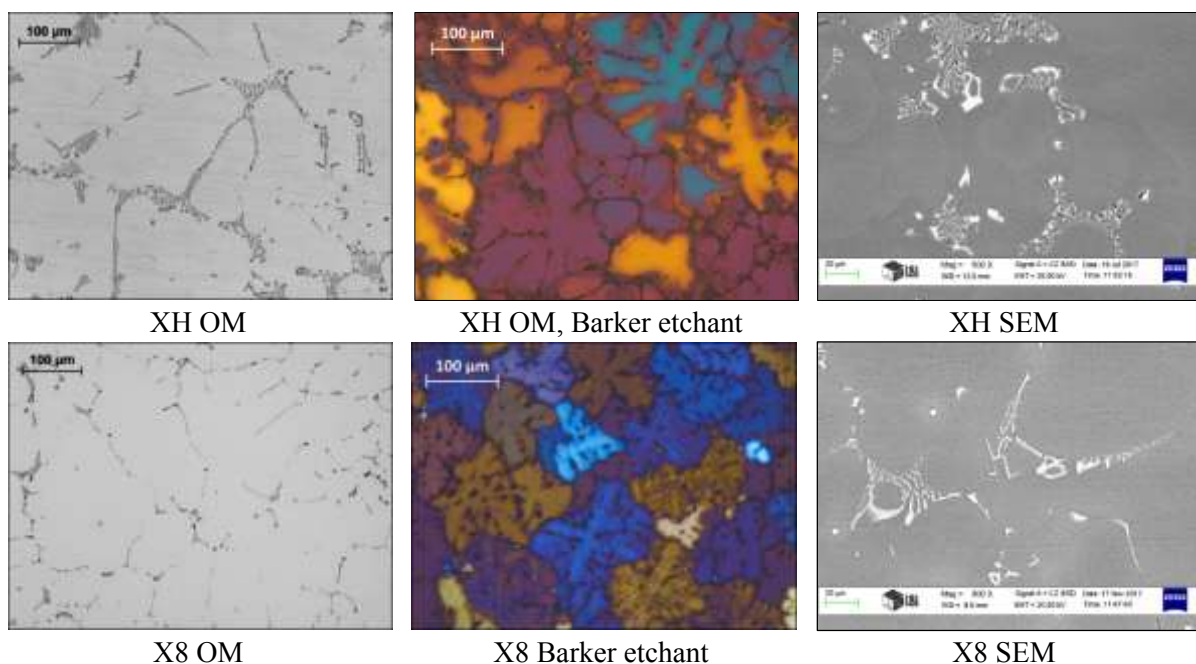


Figure 1. The as casted microstructure of the samples

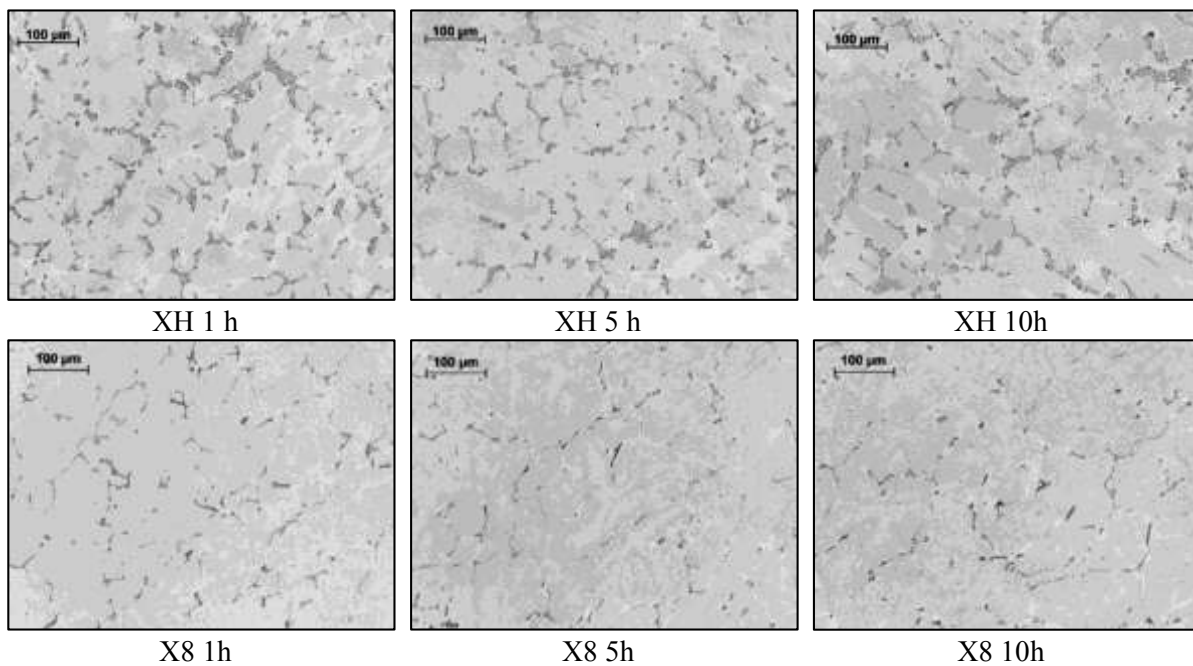


Figure 2. The light microscopy feature of homogenized sample at 510°C 1, 5 and 10 hours

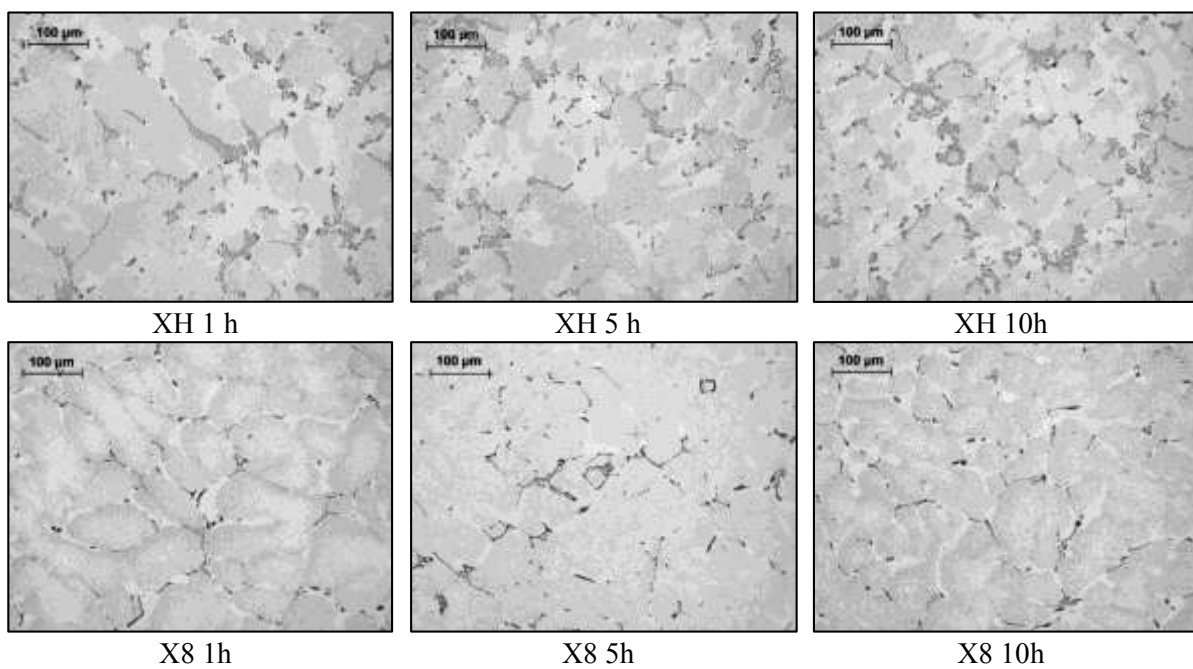


Figure 3. The light microscopy feature of homogenized sample at 540°C 4h step 510°C 1, 5 and 10 hours

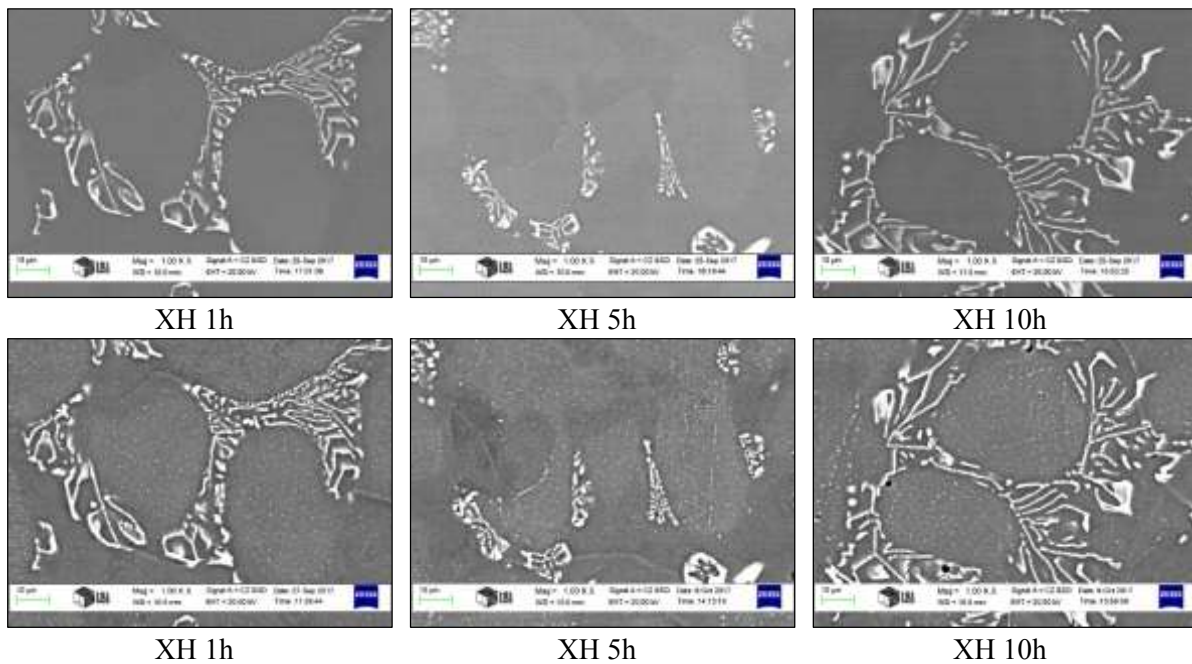


Figure 4. Correlative microscopy investigation of the XH samples before and after the homogenization

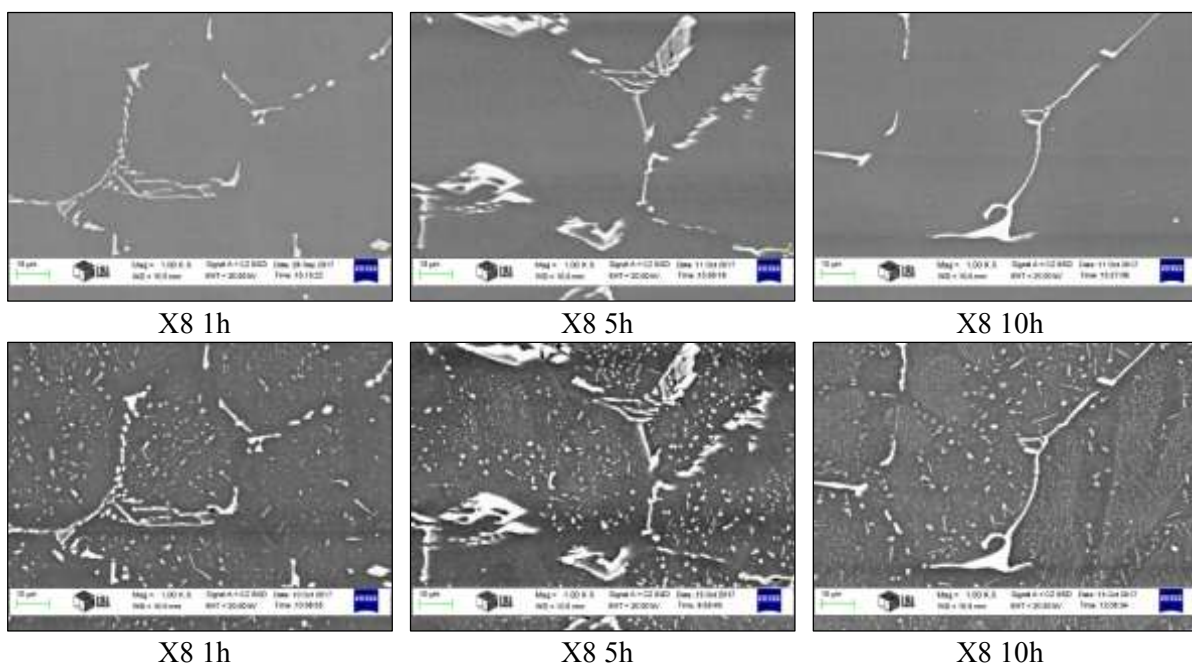


Figure 5. Correlative microscopy investigation of the X8 samples before and after the homogenization

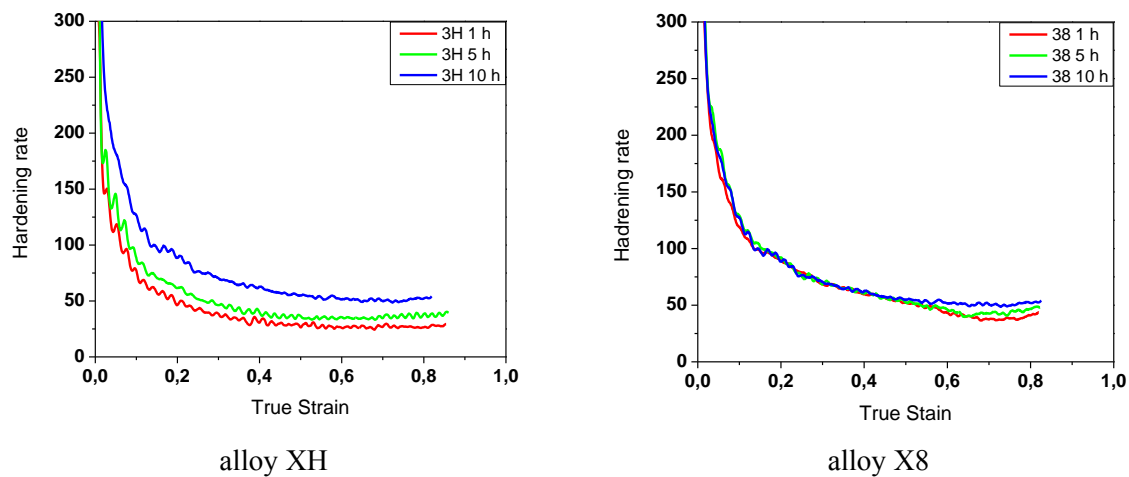


Figure 6. Hardening curves determined during cold compression test of the homogenized sample under conditions A

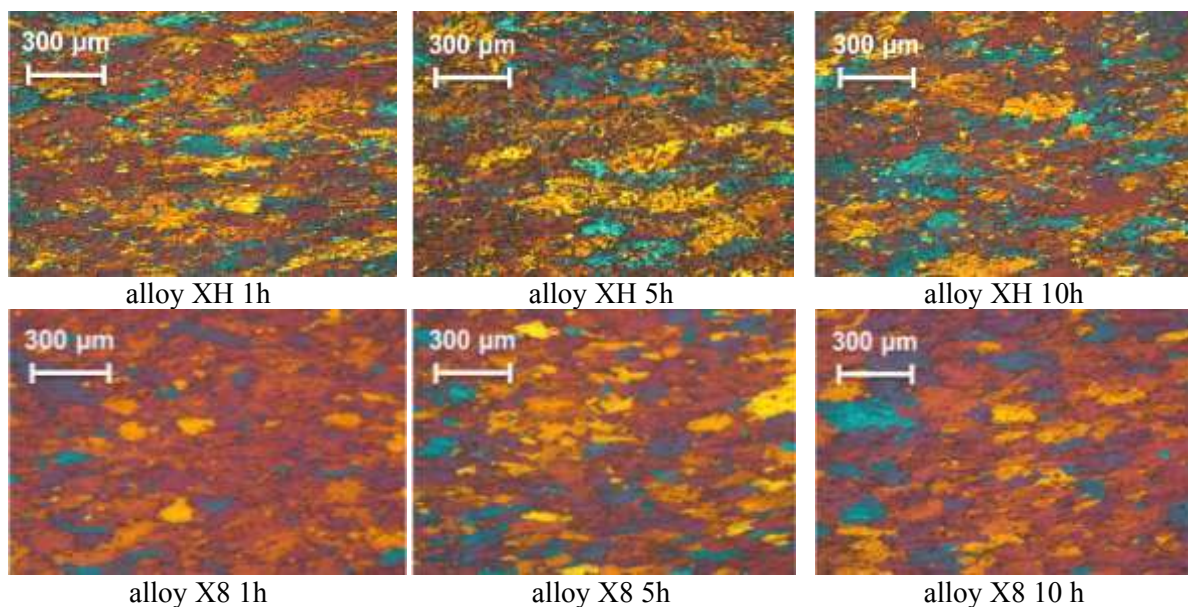


Figure 7. Microstructure of the two alloys were homogenized under condition A. After cold compression test and subsequent annealing. Barker etchant

4. Summary

Homogenization of commercial and modified 3003 aluminium alloys was investigated. The effect the increased Si, Mg, Zn were studied on the precipitation processes using different temperature and time for homogenization. Using the method of correlative microscopy, it has been clearly demonstrated that the small percentage of alloying variation causes a significant change in the microstructure. Smaller Si and Mn content causes iron-rich needle particles. In larger Si and Mn content, formation of needles is retarded and during cold deformation results in stronger hardening that increases the recrystallization tendency and may result in a finer grain size after the recrystallization process. So the dissolved elements caused hardening has stronger effect on the recrystallization than the particle stimulated process.

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References

- [1] Majed M Jaradeh R and Carlberg T 2011 *J. Mater. Sci. Technol.* 27(7) pp 615-627
- [2] Li Y J, Muggerud A M F, Olsen A and Fura T 2012 Precipitation of partially coherent α Al(Mn,Fe)Si dispersoids and their strengthening effect in AA 3003 alloy *Acta Materialia* 60 pp 1004-1014
- [3] Alexander D T L and Greer A L, 2002 Solid-state intermetallic phase transformations in 3XXX aluminium alloys, *Acta Materialia* 50 pp 2571-2583
- [4] Fujii T, Watanabe C, Nomura Y, Tanaka N and Kato M, 2001 Microstructural evolution during low cycle fatigue of a 3003 aluminum alloy, *Materials Science and Engineering*, A319-321 pp 592-596
- [5] MERCHANT H D, MORRIS J G, HODGSON D S ,1990 Characterization of intermetallics in aluminum alloy 3004, *Materials Characterization* 25 pp 339-373
- [6] Li M, Tamura T, Omura N, Murakami Y, Tada S, Miwa K and Takahashi K 2014 Refinement of intermetallic compounds and aluminum matrix in 3xxx aluminum alloys solidified by an electromagnetic vibration technique, *Journal of Alloys and Compounds* 610 pp 606-613
- [7] Miroux A, Lok Z J, Marthinsen K and van der Zwaag S, 2007 Solute and second phase evolution during industrial processing of AA3103, *Materials Science Forum* Vols. 539-543 pp 281-286
- [8] Huang H-W and Ou B-L, 2009 Evolution of precipitation during different homogenization treatments in 3003 aluminum alloy, *Material and Design* 30 pp 2685-2692
- [9] Huang H-W, Ou B-L and Tsai C-T, 2008 Effect of homogenization on recrystallization and precipitation behavior of 3003 aluminum alloy, *Materials Transactions*, Vol. 49, No.2 pp. 250 to 259
- [10] Marie A, Muggerud F, Mortzell E A, Li Y and Holmestad R, 2013 Dispersoid strengthening in AA3XXX alloys with varying Mn and Si content during annealing at low temperatures, *Materials Science & Engineering A* 567 pp 21-28
- [11] Du Q, Poole W J, Wells M A and Parson N C, 2013 Microstructure evolution during homogenization of Al-Mn-Fe-Si alloys: Modeling and experimental results, *Acta Materialia* 61 pp 4961-4973
- [12] Li Y J and Arnberg L, 2003 Quantitative study on the precipitation behavior of dispersoids in DC-cast AA3003 alloy during heating and homogenization, *Acta Materialia* 51 pp 3415-3428
- [13] Sjolstad K, Engler O, Tangen S, Marthinsen K and Nes E 2002, Texture evolution of an AA3xxx alloy after different homogenisation treatments, *Materials Science Forum* Vols. pp 396-402 pp 463-468
- [14] Liu Q, Yao Z, Godfrey A and Liu W 2009, Effect of particles on microstructural evolution during cold rolling of the aluminum alloy AA3104, *Journal of Alloys and Compounds* 482 pp 264-271
- [15] Liu W C, Zhai T and Morris J G 2004, Texture evolution of continuous cast and direct chill cast AA 3003 aluminum alloys during cold rolling, *Scripta Materialia* 51 pp 83-88
- [16] KWAG Y and MORRIS J G 1986, The effect of structure on the mechanical behavior and stretch formability of constitutionally dynamic 3000 series aluminum alloys, *Materials Science and Engineering*, 77 pp 59-74
- [17] Duan X and Sheppard T 2002, Three dimensional thermal mechanical coupled simulation during hot rolling of aluminium alloy 3003, *International Journal of Mechanical Sciences* 44 pp 2155-2172