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The Effect of Sc on the Recrystallisation Resistance and Hardness of an Extruded and Subsequently Cold Rolled Al-Mn-Mg-Zr Alloy

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Abstract

It is possible to form wrought Al alloys that are recrystallisation resistant to near incipient melting temperatures by adding Zr and Sc. In this investigation the effect of Sc on the recrystallisation resistance and hardness of a series of Al-Mn-Mg-Zr alloys was explored. The alloys were extruded, followed by cold-rolling to up to 80% reduction, and subsequently annealed 1h at temperatures in the range 300°C – 600°C. An alloy with as little as 0.11 wt.%Sc gives an almost completely non-recrystallised microstructure after annealing at 550°C, while another alloy with 0.26 wt.%Sc subjected to the same processing route remains completely non-recrystallised after annealing at 600°C. Above 400°C the hardness of the annealed material remains unaffected by the preceding cold rolling, as long as the microstructure remains non-recrystallised. After annealing at 600°C the strength contribution of the fibrous structure is only modest.

1. Introduction

Over the past 20 years there has been an increasing interest in investigating the beneficial effects that can be achieved by adding scandium to wrought aluminium alloys. Most of the reported effects are linked to the formation of particles of the Al_3Sc phase, and can roughly be divided into three groups:

1 - Grain refinement in casting or welding
2 - Zener-drag from Al_3Sc dispersoids
3 - Strengthening from Al_3Sc precipitates

Sc is well-known to be an excellent dispersoid forming element in aluminium and its effectiveness as a recrystallisation inhibitor is amplified when combined with Zr [1-3]. In a recent study of extruded Al-Mn-Mg-Zr alloys with and without Sc additions recrystallisation resistance was investigated after isothermal annealing [4]. It was found that the alloy with the highest Sc content remained non-recrystallised even after 1h at 600°C, and that this was due to the presence of $Al_3(Sc_{1-x}Zr_x)$ dispersoids. In the present investigation the potential of $Al_3(Sc_{1-x}Zr_x)$ dispersoids for recrystallisation resistance in highly deformed alloys was explored. The same alloys as in the previous study [4] were used, however, the driving force for recrystallisation was increased by cold working the as-extruded profiles.

2. Experimental Procedure

A total of five alloys was investigated, see Table 1. Alloy 1 was DC cast as Ø95mm logs and served as a baseline alloy for the Sc-containing variants Alloy 3 and Alloy 4, while Alloy 5 was made from high purity aluminium and pure binary master alloys. A limited amount of Sc was available for the investigation, therefore DC casting of the Sc containing variants was not feasible. These alloys were instead mould cast using directional solidification. This was also done for Alloy 2, which is simply Alloy 1 recast in the directional solidification mould. The alloys were homogenised by heating at a rate of 50°C/h to 470°C, keeping them there for either 4h (alloys 2, 4 and 5) or 20h (alloys 1 and 3), then quenching to room temperature in water. Extrusion billets of Ø95mm x 150mm were cut from all the alloys, preheated in an induction coil to 470°C at a rate of approx. 100°C/min and extruded to 1.9mm x 39.9mm flat bar profiles with a constant ram speed of 4 mm/s (profile exit speed approx. 415 mm/s). During extrusion the exit-temperature of the profiles was in the range 540°C-565°C, and the temperature stayed well above 500°C for the 3-4 seconds it took to hit the water-quench zone. The procedures for casting, homogenisation and extrusion are described in more detail in [4].

Table 1: Measured chemical compostions, casting method and homogenisation time of the alloys of this investigation. The composition measurements were done by an optical emission spectrograph except for the Sc-measurements, which were done by wavelength dispersive spectroscopy (WDS) in a microprobe

Alloy	Fe	Si	Mg	Mn	Zr	Sc	Casting	Hom 470°C
1	0.20	0.15	0.21	0.51	0.15	0.00	DC cast	20 h
2	0.20	0.16	0.18	0.50	0.14	0.00	Mould cast	4 h
3	0.19	0.14	0.18	0.47	0.13	0.11	Mould cast	20 h
4	0.19	0.15	0.17	0.45	0.13	0.21	Mould cast	4 h
5	0.02	0.01	0.22	0.48	0.15	0.26	Mould cast	4 h

The samples of this investigation were taken from a position approx. 2/3 of the profile runout length. A sample from each alloy was kept in the as-extruded condition, while four other samples were cold rolled at room temperature to nominal reductions of 5%, 20%, 50% and 80%. The measured thicknesses of the as-extruded samples and cold rolled samples are listed in Table 2, along with the actual reduction. The samples were then cut to smaller pieces, and subjected to 1 h annealing at either 300°C, 350°C, 400°C, 450°C, 500°C, 550°C or 600°C in an air circulation furnace. The samples reached the furnace temperature within approx. 5 min. Optical light microscopy observations were done on polished and anodised samples of the longitudinal cross sections at approx 10 mm from the profile edge. The fraction of recrystallised grain structure in the samples was determined by the linear intercept method across the specimen thickness. The Vickers hardness was measured in mid-thickness of a similar cross section as that which was studied in the light microscope. A load of only 0.3 kg was applied due to the small specimen thickness of the thinnest samples.

Table 2: Measured thicknesses and actual reduction in % of the samples

Alloy	As-extr.	5% reduction		20% reduction		50% reduction		80% reduction	
	mm	mm	act. %	mm	act. %	mm	act. %	mm	act. %
1	1.81	1.63	9.9	1.43	21	0.90	50	0.32	82
2	1.81	1.63	9.9	1.42	22	0.88	51	0.33	82
3	1.79	1.64	8.4	1.45	19	0.90	50	0.34	81
4	1.78	1.66	6.7	1.47	17	0.93	48	0.36	80
5	1.77	1.66	6.2	1.47	17	0.92	48	0.36	80

3. Results and Discussion

Figure 1 shows the results from the hardness measurements and the fraction of recrystallised grain structure as determined from the optical light microscope observations. Figure 2 shows some examples of the microstructures of the alloys. For Alloy 1 and Alloy 2 the specimens annealed at 450°C are shown, while the specimens annealed at 600°C are shown for Alloy 3, Alloy 4 and Alloy 5.

There is a notable increase in recrystallisation resistance when Sc is added to the alloys. Alloy 3, with as little as 0.11 wt.%Sc, gives an almost completely non-recrystallised microstructure after extrusion and 80% cold-rolling with subsequent annealing at 550°C. Alloy 5, with 0.26 wt.%Sc, subjected to the same process route remains completely non-recrystallised after annealing at 600°C.

From Figure 1 one finds that Alloy 1 and Alloy 2 have an almost similar development of hardness and fraction of recrystallised grain structure as a function of annealing temperature. The main difference is that the 50% and 80% cold rolled specimens recrystallise at approx. 50°C lower temperatures for Alloy 1 than for Alloy 2. The recrystallisation is accompanied by a hardness decrease. Another noticeable difference between Alloy 1 and Alloy 2 is the development of a thicker recrystallised surface layer in Alloy 2 compared to Alloy 1, see Figure 2. The differences in recrystallisation behaviour may be due to differences in homogenisation heat treatments that were applied, and thus differences in the dispersoid spatial density and size in the alloys. Differences in initial microstructures, due to different casting practices between Alloy 1 and 2, may have also led to different distributions of accumulated plastic deformation across the profiles, and hence different profiles of recrystallisation driving force for those alloys.

In Alloy 3, Alloy 4 and Alloy 5 it is evident that the addition of Sc imparted higher hardness as well as a much higher recrystallisation resistance compared to Alloy 1 and Alloy 2. The hardness of the alloys increases with increasing Sc content. After annealing at temperatures lower than 400°C – 450°C the hardness also increases with increasing degree of preceding cold rolling of the material. For the Sc-containing alloys the increase in hardness due cold-rolling is gradually annealed out with increasing annealing temperature, and after annealing above 400°C – 450°C there is no increase in hardness due to the preceding cold rolling, as long as the alloy remains non-recrystallised.

One notices that for all the investigated alloys, the hardness of the as-extruded and subsequently annealed samples remains at the same level up to annealing temperatures of 400°C-500°C. It could be that 3-4 seconds of exposure to high temperature after extrusion, *i.e.* between the die exit and the quench zone, gives a microstructure that does not recover further upon annealing in the range 300°C – 400°C. However, it could also be that the Mn and/or Sc that is brought into solid solution during extrusion re-precipitates at the lower annealing temperatures and that such a re-precipitation counterbalances the hardness drop that should follow from recovery processes of the dislocation- and subgrain structure. A TEM-investigation would reveal whether one of these hypotheses, or both, are valid.

The stabilising $Al_3(Sc_{1-x}Zr_x)$ dispersoids in the Sc-containing alloys were formed during homogenisation at 470°C. It is not anticipated that the 1h annealing of the extruded and cold rolled samples at temperatures of 450°C and below leads to any appreciable coarsening of these dispersoids.

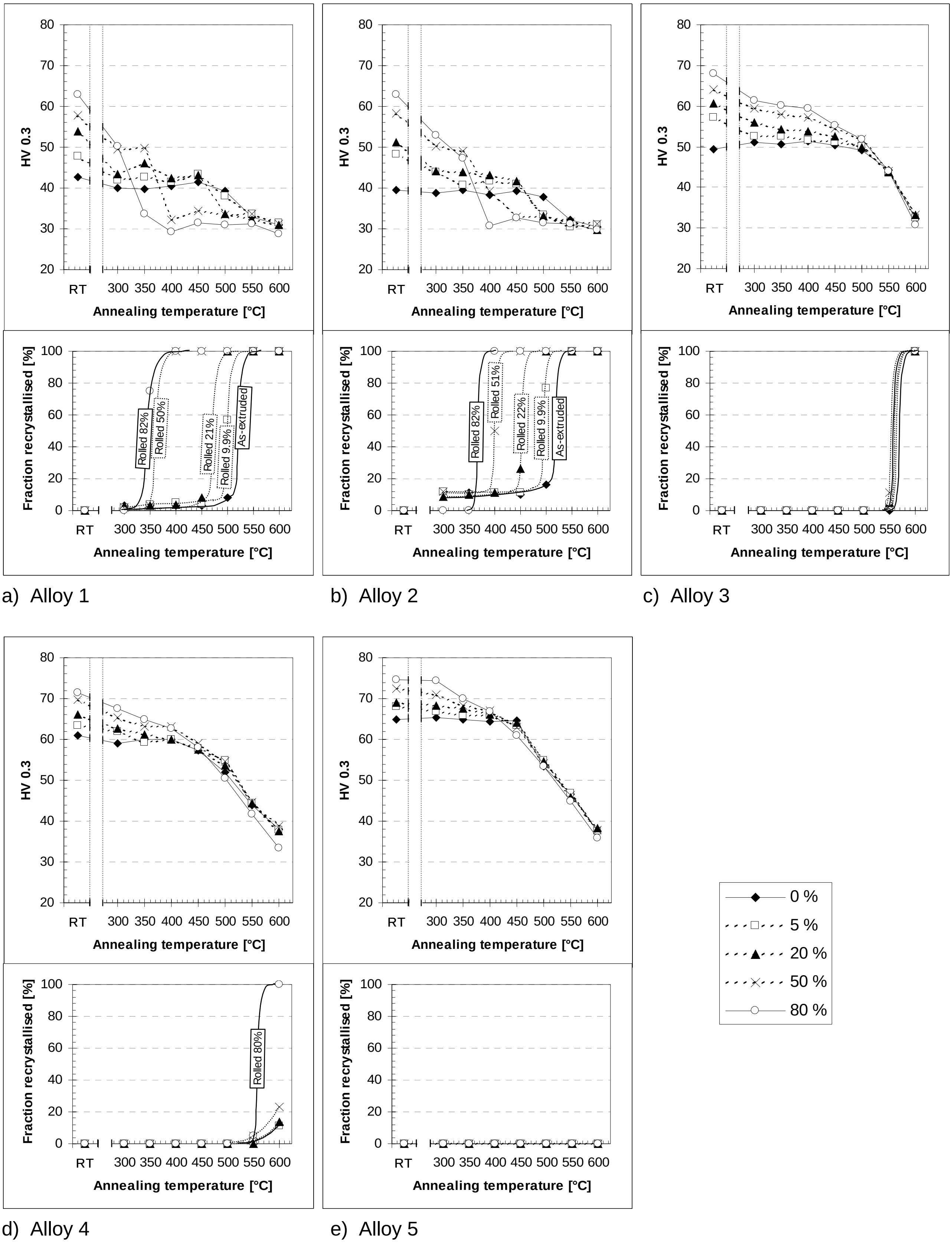
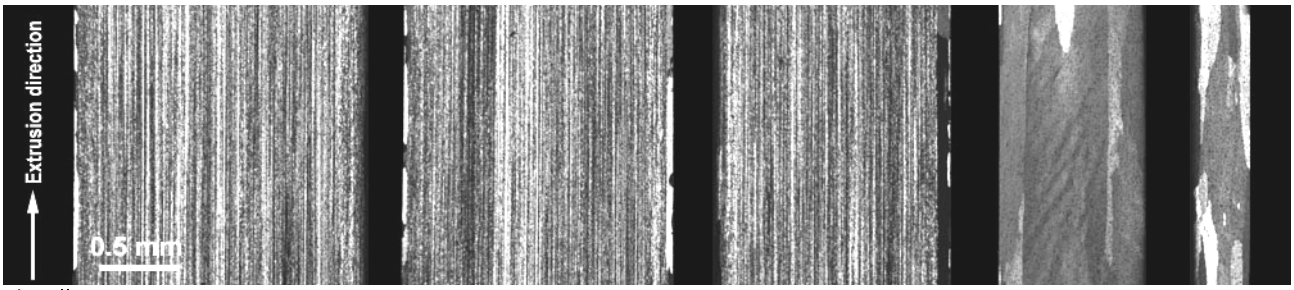
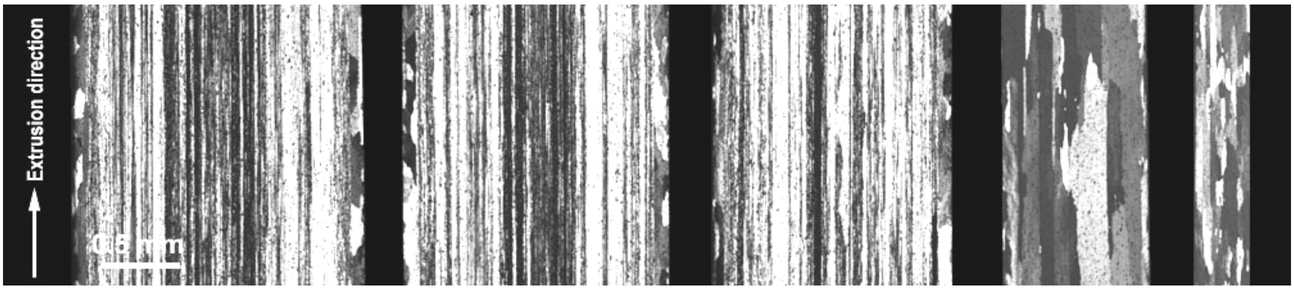


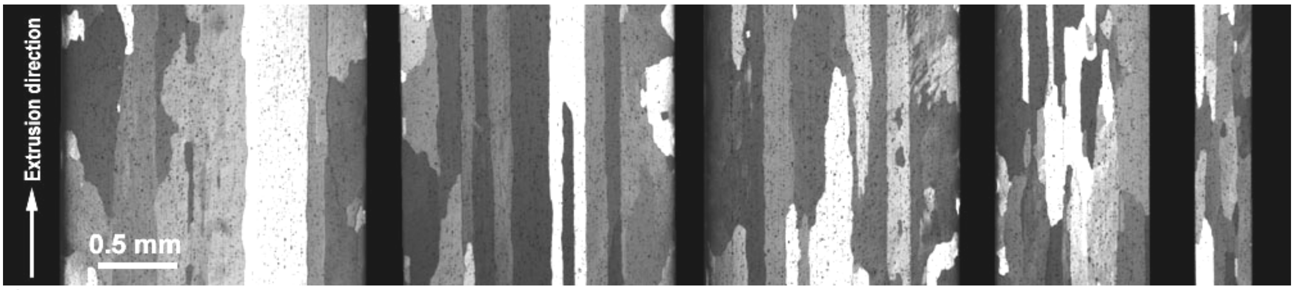
Figure 1: Hardness measurements and measured fraction recrystallised material as a function of degree of cold rolling and subsequent annealing temperature for the investigated alloys. The legend (bottom right) indicates the nominal degree of cold rolling of the specimens.



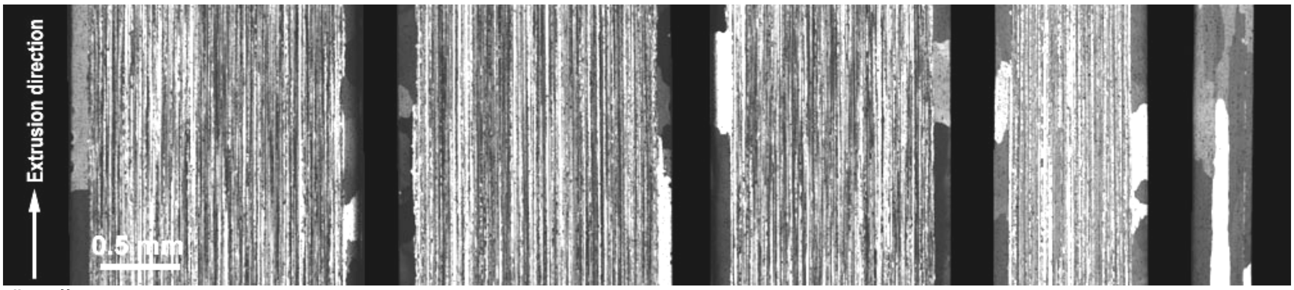
a) Alloy 1, 450°C



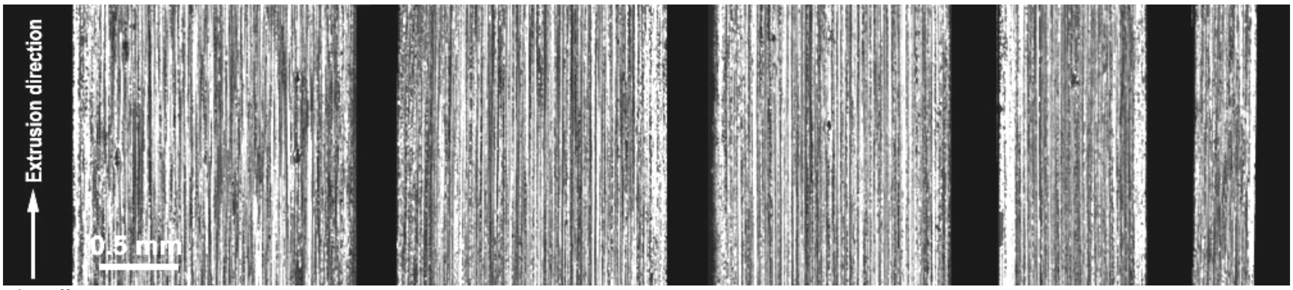
b) Alloy 2, 450°C



c) Alloy 3, 600°C



d) Alloy 4, 600°C



e) Alloy 5, 600°C

Figure 2: Examples of microstructures as observed in the optical light microscope. For each alloy, the specimens are ordered from left to right with increasing degree of cold rolling. For Alloy 1 and Alloy 2 the specimens annealed at 450°C are shown, while the specimens annealed at 600°C are shown for Alloy 3, Alloy 4 and Alloy 5.

However, the 1h annealing at 500°C, 550°C and 600°C is expected to lead to considerable coarsening as well as partial dissolution of the dispersoids. The coarsening rate increases with the increasing temperature. At the same time an increasing amount of Sc and Zr is brought into solid solution, thus leading to a decrease in the available volume fraction of dispersoids. The impact of these processes on the hardness of the alloys is twofold. First, the contribution on the hardness from the dispersoids themselves is reduced due to their reduced number density and volume fraction. Secondly, the lower Zener-drag force from the coarsening and dissolving dispersoids leads to a faster recovery of the dislocation- and subgrain-structure. A detailed TEM-investigation would be necessary in order to quantify how much of the hardness drop that is associated with each of these two softening mechanisms.

Looking at the hardness values of Alloy 4 specimens annealed at 600°C, the 80% cold-rolled specimen has a somewhat lower hardness compared to less deformed specimens. In the light microscope (Figure 2) one finds that the 80% specimen is recrystallised while the other specimens are non-recrystallised in the area where the hardness was measured. Thus, considering the similar hardness evolution of the non-recrystallised specimens, this hardness difference serves as a measure of the contribution of the fibrous structure to the overall strength of the alloy for this particular annealing temperature and time. As one can see, after annealing at this high temperature the strength contribution of the fibrous structure is modest.

4. Conclusion

$Al_3(Sc_{1-x}Zr_x)$ dispersoids greatly increase the recrystallisation resistance of highly deformed Al-Mn-Mg-Zr alloys compared with non-Sc containing variants. An Al-Mn-Mg-Zr alloy with as little as 0.11 wt.%Sc gives an almost completely non-recrystallised microstructure after extrusion and 80% cold-rolling with subsequent annealing at 550°C. When the content of Sc is raised to 0.26 wt.% and the alloy subjected to a similar thermomechanical process route the microstructure remains completely non-recrystallised after annealing at 600°C. At annealing temperatures of approx. 400°C – 450°C and above there is no effect of the preceding cold rolling on the hardness of the annealed material as long as it remains non-recrystallised. After annealing at 600°C the strength contribution of the fibrous structure is modest. Incorporation of $Al_3(Sc_{1-x}Zr_x)$ dispersoids should be considered a more effective route for developing high recrystallisation resistance in Al alloys compared to the use of either Al_3Zr or Al_3Sc dispersoids alone.

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References

[1] V.I. Yelagin, V.V. Zakharov, S.G. Pavlenko and T.D. Rostova, Phys. Met. Metall., 60, pp 88-92. 1985
[2] V.G. Davydov, V.I. Elagin, V.V. Zakharov and T.D. Rostova, Met. Sci, Heat Tr., 38, pp 347-352, 1996
[3] Y.W.Riddle and T.H. Sanders, Jr., Met. Mater. Trans. A, 35A, pp 341-350, 2004
[4] Y.W. Riddle, H. Hallem and N. Ryum, Mater. Sci. Forum, 396-402, pp 563-568, 2002