STRATEGIES TO ELIMINATE GRAIN COARSENING IN AEROSPACE ALLOYS

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ABSTRACT

AMAG investigated different aspects to improve the resistance against grain coarsening in aerospace alloys during recrystallization and was successful in finding several opportunities to minimize this effect. The studies show that one aspect to counteract grain coarsening is the chemical composition and subsequently all phases that are formed during homogenization. Starting with a fine grain (~ 50 µm) can lead to a final grain size of few mm. On the other hand with a coarse initial grain size (approximately 150 µm) and the right chemical composition the grain size can stay almost constant despite different deformation steps. Additional we investigated different sequences for the component production to eliminate the problem of grain coarsening as a coarse grain has a detrimental effect on product performance.

KEYWORDS

2024 aluminium alloy, Grain coarsening, Recrystallization, W-temper forming

INTRODUCTION

In the aircraft industry, aluminium sheets are most commonly deformed in their soft state, followed by solution heat treatment and natural or artificial aging. If the preceding deformation is in a critical amount, during the recrystallization at solution heat treatment grain coarsening can occur. A subsequent deformation to this condition leads to an orange peel surface, which requires additional polishing. Next to this extra effort the coarse grain size produces a drop in the mechanical properties, often below specification limits. If there is no subsequent deformation, this effect is not visible and without destructive testing there is no possibility to detect the error. As mentioned before, this effect occurs during recrystallization, which is a process that is driven by the stored energy in the microstructure.

The two most important aspects for an increase of the stored energy are the increase of the grain boundaries fraction (smaller grain size) and the increase of the dislocation density caused by deformation. The increase in dislocation density during the deformation arise from trapping of existing dislocations and from the generation of new dislocations. During recrystallization all grains are built completely new, so all dislocations are removed (Humphreys & Hatherly, 2004). There are three requirements for recrystallization:

- Thermodynamic instability: nuclei need a minimum size
- Mechanical instability: the dislocation distribution has to be inhomogeneous, otherwise the driving force is constant for the whole material and there is no power for recrystallization
- Kinetic instability: the grain boundaries of the nucleus must be moveable (Zurob et al., 2006)

A successful nucleus is growing constantly until it meets another newly formed grain and the deformed material is completely recrystallized (Gottstein, 2007).

In the present study we investigated the influence of the grain size, chemical composition and two different sequences for the component production.

EXPERIMENTAL METHODS

Material

For all experiments we used a 2024 aluminium alloy with a thickness of 1.6 mm. The average chemical composition of the main elements is listed in Table 1. Materials A and B have slightly more impurities than material C, the main elements are in the same composition. We prepared the alloys in an electromagnetic cast furnace, homogenized them with air cooling afterwards. Subsequently we hot rolled, cold rolled and heat treated the samples in different procedures to realize different grain sizes and different tempers. Initial grain size and temper are listed in Table 2. To improve the corrosion resistance of 2xxx aluminium alloys, the material is cladded with 4 to 5% pure aluminium.

Table 1. Average chemical composition (%) of the main elements

Cu	Mg	Mn	Zn
4.4	1.5	0.5	0.15

Table 2. initial grain size, initial temper (untreated samples) and test sequences

Material	grain size [μm]	Temper	Thickness [mm]	Different composition	Test sequence
A	~ 50	O	1.6	Yes	deformation → SHT → tensile test
A1	~ 50	O	1.6	Yes	SHT → deformation (within 30min) → tensile test
B	~ 150	O	1.6	Yes	deformation → SHT → tensile test
B1	~ 150	O	1.6	Yes	SHT → deformation (within 30min) → tensile test
C	~ 30	O	1.6	No	deformation → SHT → tensile test

Test Methods

To realise different deformation steps, we deformed the samples under uniaxial tension from 2 to 14% elongation in the soft state. Subsequently we solution heat treated (SHT) the samples at 493°C for 20 min in a salt bath to initiate recrystallization. We deformed Material A1 and B1 under uniaxial tension within 30 minutes after SHT to simulate W-Temper forming. All test procedures are listed in Table 2. To evaluate the strength and ductility of the samples, we carried out tensile tests at room temperature. The test set up was in accordance with EN ISO 6892-1. To calculate the grain size, we manufactured metallographic samples in cross section and applied the linear intercept procedure according to ASTM E112-13. It has to be mentioned that variability is less than ± 0.5 ASTM grain size, which means ± 10 μm for an average grain size of 50 μm.

RESULTS AND DISCUSSION

Critical Deformation

During deformation dislocations move in the material, they are multiplied and accumulated at impurities and grain boundaries. Areas with accumulated dislocations are areas with high energy and consequently preferred nucleation points. At a critical degree of deformation there are only a few successful nuclei in the material which can grow until they meet, in addition the final gain size gets coarse. With further deformation more successful nuclei are created and the final grain size gets smaller. This effect is shown in Figure 1. Material A was bent through 180° with a radius of 5 mm and subsequently SHT at 493°C for 20 min. The difference in the deformation of the peripheral zone and neutral fibre can be seen in the final grain size. In the peripheral zone the deformation seems to be in the order of the critical deformation degree so the grain size after recrystallization is big. In the neutral fibre the grain size before and after the recrystallization is similar, so there are no preferred recrystallization points.

before SHT		after deformation and SHT

Figure 1. critical degree of deformation of Material A

Another visual representation is shown in Figure 2. Material C was deformed and SHT as described in section experimental methods. Depending on the deformation degree the final grain size is different. In this material the abnormal coarse grain starts at 6% deformation (grain size > 2,000 μm), at 9% deformation the grain size is approximately 400 μm. The critical deformation degree and the impact to the final grain size can be influenced with the initial grain size and the chemical composition (Drossel et al., 1996; Kammer, 2002).

| 0% prestrain to SHT | 2% prestrain to SHT | 4% prestrain to SHT |
| 6% prestrain to SHT | 8% prestrain to SHT | 9% prestrain to SHT |
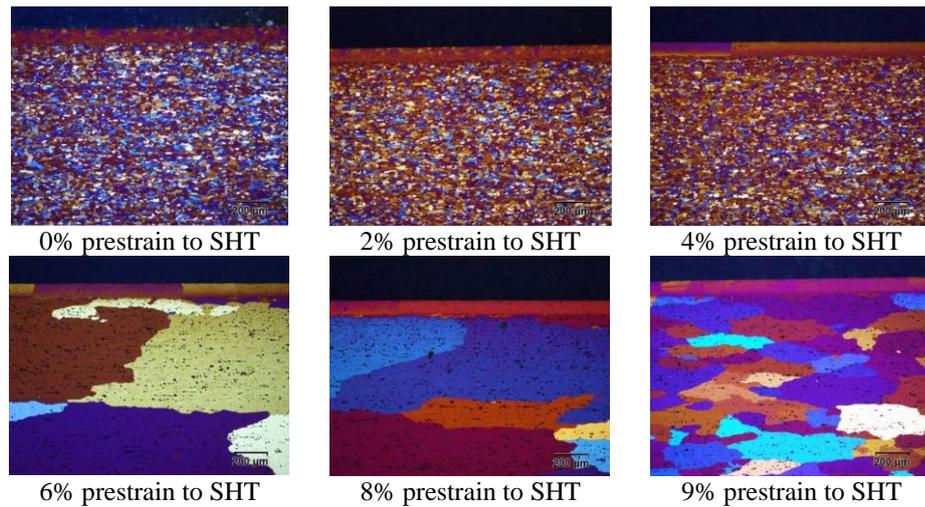
Figure 2. Material C with different prestrain to SHT

Initial Grain Size

Our results show a strong dependence between the initial grain size and the resistivity to grain coarsening. Starting point and extent of the grain coarsening are affected by the initial grain size. Dislocations accumulate in front of grain boundaries because they cannot overcome them. In material with a small grain size the dislocations have only a small distance where they can move without accumulation. This unbalanced energy distribution occurs in small grain size material at a low deformation degree. At subsequent heat treatment only a few successful recrystallization nucleons are induced and the final grain size is big. In material with a big initial grain size the energy is spread more homogenously, subsequently a higher degree of deformation is needed for a high enough energy gradient for recrystallization. As a result more nuclei are formed simultaneously (Neuhaus, 2013).

This aspect can be seen in Figure 3. Material A with an initial fine grain size (~ 40 µm) has a critical deformation degree at about 9%. Material B with initial coarse grain (~ 150 µm) has a critical degree of deformation at 10%. Additionally, the biggest grain size in material B is about 1300 µm compared to a maximum grain size of approximately 1800 µm in material A.
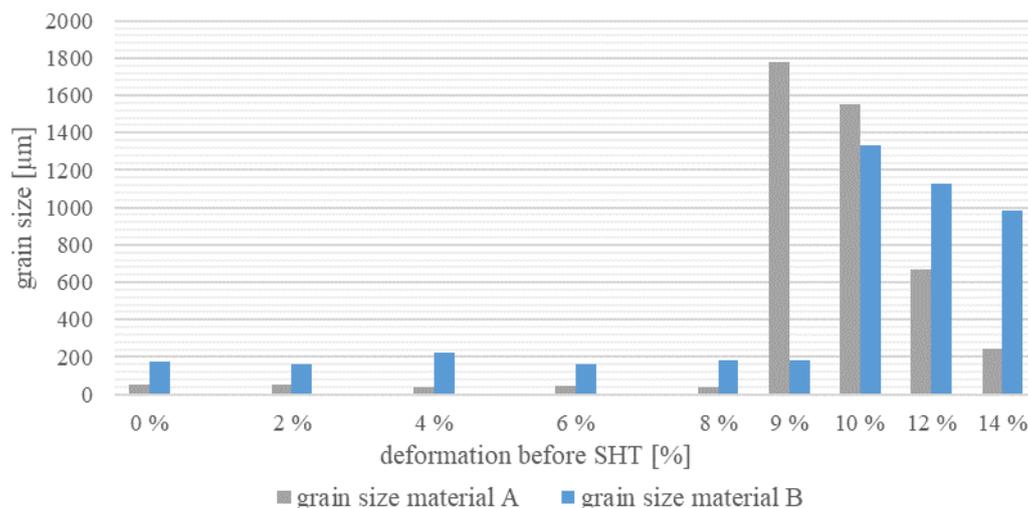
Figure 3. Grain size of material A and B after deformation and SHT

Chemical Composition

There are two aspects how the chemical composition can influence the critical degree of deformation and the final grain size. First, the alloying elements reduce the stacking fault energy, therefore the dislocations can split easily and are less mobile any more. As a further consequence the dislocation cell formation takes place to a smaller extend, which means that the dislocation density is more homogeneous and for recrystallization nucleation a higher degree of deformation is needed. This phenomenon can be observed in the metallographic sections in Figure 4, where the grain coarsening in the cladding layer occurs earlier than in the bulk material (Ostermann, 2007; Vasudevan & Doherty, 1989).
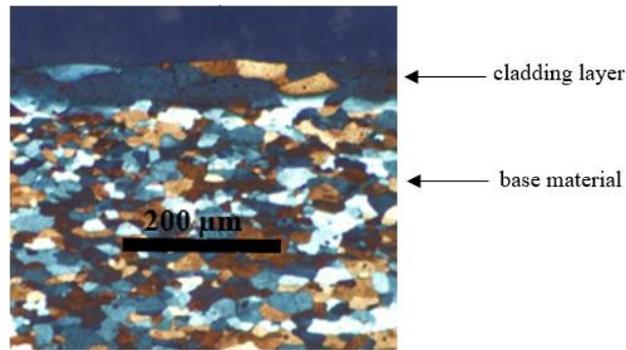
Figure 4. Different grain size of base material and cladding layer (material B1)

Secondly the chemical composition influences which phases are built and in addition the phases influence the final grain size. Depending on their size phases can act as dislocation multiplier or they pin the grain boundaries and inhibit grain coarsening. Both effects result in a smaller final grain size. Phases which arise out of impurities are mostly very small and act as grain boundary restrainer (Vasudevan & Doherty 1989; Hatch, 1984). This phenomenon can be seen in Figure 5. We deformed Material A and Material C different degrees and carried out a subsequent SHT. The material with higher content of impurities (material A) shows a higher resistivity against grain coarsening. The grain coarsening takes places at a higher degree of deformation and additionally to a lower extend.
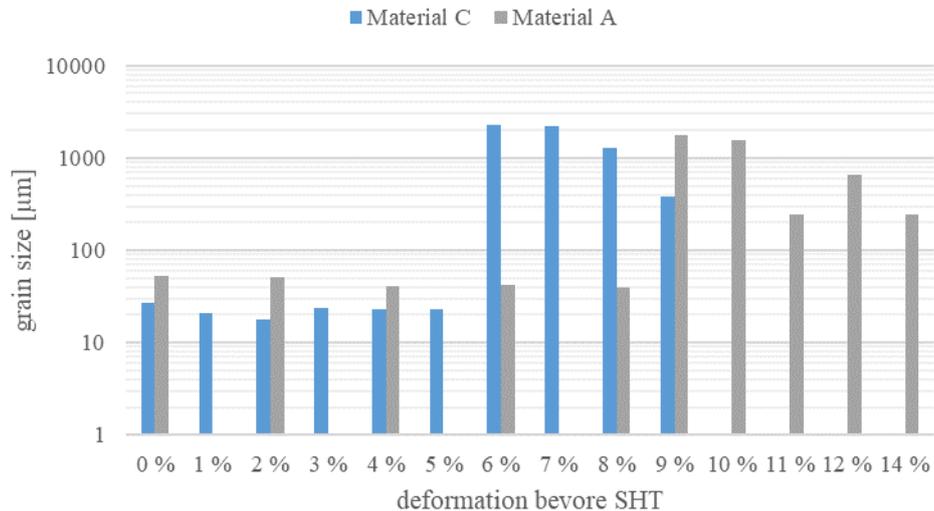
Figure 5. Grain size of material A (with impurities) and material C (without impurities)

To determine the cause of the higher resistivity against grain coarsening in material with higher impurity content both materials were investigated in high resolution scanning electron microscope (HRSEM) and transmission electron microscope (TEM). There are some phases (dispersoids) which pin the grain boundaries and therefore retard the recrystallization (Figure 6). Quantitative analysis in TEM show that the material with high impurity content contains more of these phases (Table 3)

Figure 6. Dispersoid pins a grain boundary

Table 3. Frequency of disperoid in material with high and low impurity content

	Frequency of dispersoids	
	$[1/\mu m^2]$	$\pm [1/\mu m^2]$
Low impurity content	3,9	1,1
High impurity content	6,3	0,9

Different Sequences in the Production Route

Usually aluminium sheet material is deformed in its soft state, followed by SHT. Recrystallization during SHT often results in a different grain size which is detrimental for the mechanical properties and the surface appearance. A deformation in common temper T4 leads to a very small elongation at break leading usually to material failure. This process route is therefore unusual. However during a short period after SHT the material has a low strength and a high elongation at break. Having a low degree of natural aging a high degree of deformation is achievable. Figure 7 (left) shows material that is formed in soft state with subsequent SHT. On the right side the same material has undergone SHT and was subsequently deformed within 30 minutes after SHT. During this time the material is soft enough for deformation. The advantage of this production route is that there is no recrystallization after deformation and thus there is no difference in the grain size between different deformation degrees.
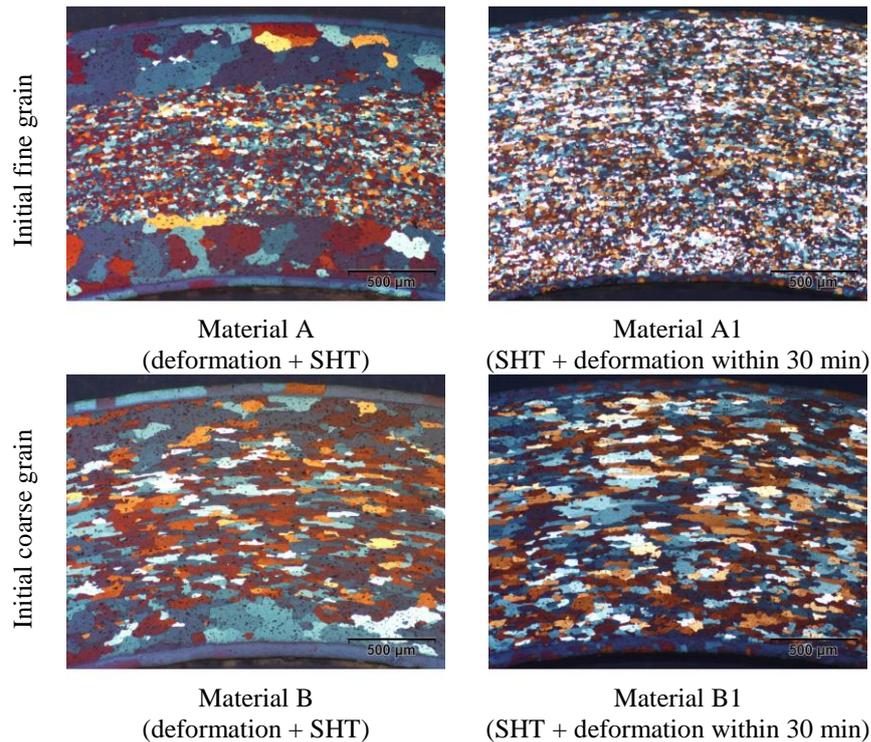
Figure 7. Influence of the production route to the final grain size

CONCLUSIONS

An equal distribution of the final grain size in the production is desirable for a uniform strength. In this paper the most important aspects which should be noted for an equal distribution of the grain size after deformation and recrystallization were discussed.

(1) A big (~ 150 μm) initial grain size should be preferred.
(2) The deformation should exceed a minimum critical degree (15%).
(3) A small amount of impurities promotes an equal distribution of the grain size.
(4) W-temper forming should be preferred.

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