Plasticity in Aluminum Alloys at Various Length Scales
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Chapter 4

Aluminum-Scandium Alloys

Aluminum can be alloyed to meet the requirements set by quite diverse applications. The alloy strength however, is always considerably lower than that of comparable steels. Increasing the alloys strength, implies creating microstructural obstacles for dislocation motion in the material. In the binary aluminum-scandium system precipitation hardening causes a large increase in yield strength with only minute scandium additions. In this chapter this system is studied in depth. The theory of precipitation hardening as described in the previous chapter is applied in an attempt to explain the hardening characteristics of the alloy.

4.1 Introduction

In the 19th century, the little village of Ytterby near Stockholm, Sweden played a remarkably large role in the quest for new elements. From the local mine, the mineral Ytterite was extracted, which was later renamed Gadolinite in honor of the Finnish chemist Johan Gadolin. From this mineral a long list of new earths was discovered, which in those days was regarded being equivalent to the discovery of an element. With an apparent lack of imagination, four of these new elements were even named after the village (Ytterbium, Yttrium, Erbium and Terbium). In 1871, Uppsala professor Lars Fredrik Nilson, still studying the Gadolinite mineral, managed to split the earth ytterbia into a new ytterbia and a new element, which he named scandium after his homeland Scandinavia [1]. In the meantime Mendeleev had introduced his periodic table of elements and Nilson was able to show that the properties of his scandium matched exactly those of eka-boron, a missing element predicted by Mendeleev.
Scandium is widely dispersed in minute quantities in the Earth’s crust. Only the mineral Thorite possesses a significant quantity of scandium oxide (30-40%). Consequently, scandium is not generally mined economically, but it is extracted mainly as a byproduct of iron, uranium or tantalum mining. The first pound of pure scandium was not produced until 1960. Due to its costs, applications of scandium were at first limited. It is used in mercury vapor lamps to imitate natural sunlight and has applications in laser research and aerospace technology.

The demand for scandium increased however, when in 1971 Willey patented the first aluminum-scandium alloy [2]. He found that adding minute quantities (0.1-1.0%) of scandium to a number of aluminum alloys, significantly improved their mechanical properties. Al-Sc alloys are characterized as having superior corrosion resistance and weldability; they are strong, light weight and very stable at elevated temperatures. Furthermore Al-Sc alloys turned out to be well suited for superplastic forming operations. Nowadays the alloys are used for instance to make sporting equipment that has to be light weight and very strong, e.g. bicycle frames and baseball bats. There is a growing interest from industry, but the price of aluminum-scandium master alloys is still too high for most commercial applications. There are plans however, to extend existing Bayer process plants, which extract alumina from bauxite, to produce also scandium oxide [3]. This would reduce the scandium costs enormously and it is thus expected that scandium will be used much more extensively in the forthcoming years.

This chapter aims at understanding the effects of scandium additions to aluminum. The first section will deal with the microstructural examination of the alloy and in the second part, the effect of the microstructure on the mobility of dislocations will be studied. Finally, the ternary Al-Mg-Sc alloy will be treated, which has some very interesting properties due to the complementary effects of the scandium and magnesium additions.

4.2 Microstructure of an Al-0.2wt.%Sc alloy

No element other than scandium is, with the same atomic fraction, such an impressive strengthen of aluminum. To understand the large increase in yield strength after proper alloying and heat treatment, accurate knowledge of the microstructure is essential. The binary Al-Sc phase diagram is a good starting point.
4.2.1 Phase diagram

Figure 4.1 shows the binary Al-Sc phase diagram [4]. The first study of the entire composition range was done by Naumkin et.al. [5]. They showed that in this system four compounds exist: Al$_3$Sc, Al$_2$Sc, AlSc and AlSc$_2$, all with narrow homogeneity ranges. At the aluminum-rich end, a peritectic was reported at 665 °C, but later studies demonstrated that this was incorrect [6-9]. As can be seen in the aluminum-rich end of the phase diagram shown in Figure 4.2, Al and Al$_3$Sc form eutectically from the melt:

\[ L \rightleftharpoons \alpha(Al) + Al_3Sc \]  

(4.1)

The reported eutectic temperature varies between 655 and 659 °C and the scandium concentration at the eutectic is approximately 0.6 wt.% [3]. The maximum solubility of scandium in aluminum is approximately 0.35 wt.%. This already very low solubility drops rapidly upon cooling. At 500 °C only 0.075 wt.% scandium dissolves in the aluminum matrix [7].
4.2.2 Material and heat treatment

For the experiments described in this chapter a cast binary Al-0.2wt.%Sc alloy is used for which the composition is given in table 4.1.

Table 4.1 Composition of Al-0.2wt.%Sc alloy

<table>
<thead>
<tr>
<th>element</th>
<th>Al</th>
<th>Sc</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>content (wt.%)</td>
<td>99.7</td>
<td>0.19</td>
<td>0.001</td>
<td>0.03</td>
<td>0.07</td>
<td>&lt;0.005</td>
</tr>
</tbody>
</table>

Due to the difference in cooling rate, the solidification characteristics vary from the bottom to the top of the cast. This can have a large influence on the resulting microstructure. Therefore, an EDS analysis of the as-cast alloy has been performed, which shows that the composition is fairly homogeneous throughout the cast. Nevertheless, to ensure optimal homogeneity, especially of scandium, the alloy has been subjected to a homogenization heat treatment for 24 hours at 640 °C. This temperature is extremely high for an aluminum alloy, which melts at 660 °C. The treatment is however necessary in order to avoid the rapid formation of the Al₃Sc phase. According to [3], the minimum temperature at which 0.19 wt.% scandium dissolves completely in aluminum is approximately 600 °C. Although the risk of some small Al₃Sc formation exists, because of temperature fluctuations, this
approach is preferred above the risk of local melting, which would be extremely detrimental to the mechanical properties of the material.

The concentrations of impurity elements iron and silicon are relatively high, as a consequence of the use of commercially pure aluminum, but they tend to form intermetallic compounds at grain boundaries (Fig. 4.3), which are not believed to influence the overall mechanical properties of the alloy very much. Furthermore, in all TEM samples studied, only occasionally an intermetallic particle has been observed and the concentration of impurities in solid solution in the aluminum matrix as measured by EDS is negligibly low. This indicates that most impurities are tied up with intermetallics upon solidification and are not influenced much by further heat treatments.

Figure 4.3  SEM picture of homogenized microstructure. Some grain contrast can be seen, indicating a large grain size. Intermetallic particles are present throughout the sample, but have a tendency to form at grain boundaries.

After homogenization, the grain size has become extremely large due to the long time at a very high temperature and the low number of grain refining particles. These millimeter sized grains are not very practical in a TEM analysis of grains, because in the entire electron transparent area only one orientation may be present. The grain size is reduced by performing one or two cold rolling treatments, during which the thickness of the material is reduced by 70%. This reduction is achieved by rolling in small steps. After each step the material is rotated in order to avoid the development of a texture. The reduction is followed by a recrystallization heat treatment for ten minutes at 640 °C. Again, this high temperature is to avoid the formation of the Al₃Sc phase. The resulting grains are still large (~200 µm) but significantly smaller
than after homogenization. When the sample is taken out of the oven, it is immediately quenched in cold water to create a supersaturated solid solution. After recrystallization, all scandium is assumed to be in solid solution and the material is free of defects. To strengthen the material it has to be aged as explained in chapter 3. Upon aging the Al₃Sc phase will grow. Since diffusion of scandium in aluminum is slow, aging has to be performed at temperatures that are relatively high compared to other aluminum alloys. For this research aging has been performed at 300, 350 and 400 °C for periods of time ranging from 10 minutes to 96 hours. To study precipitation in a deformed matrix, one set of samples has been aged immediately after the final rolling treatment, thus without a final recrystallization. After the aging treatment the samples are air cooled instead of water quenched to relax the stresses that arise from cooling.

![Atomic arrangement of the Al₃Sc phase. This L₁₂ ordered structure is very similar to an fcc lattice but is in fact simple cubic with a four atom base.](image)

**Figure 4.4** *Atomic arrangement of the Al₃Sc phase. This L₁₂ ordered structure is very similar to an fcc lattice but is in fact simple cubic with a four atom base.*

### 4.2.3 Al₃Sc precipitates

When aged from the supersaturated solid solution, a fairly simple two phase system develops. The α(Al) phase can, as a consequence of the extremely low scandium solubility at moderate temperatures, be considered to behave as pure aluminum. Therefore, it is apparent that the hardening observed in aluminum-scandium alloys is caused by the formation of Al₃Sc precipitates.
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Figure 4.5  A [001] diffraction pattern from Al\textsubscript{3}Sc precipitates aged for 24 hours at 350 °C in an aluminum matrix. The superlattice reflections are clearly visible.

Crystallographic structure

The first to describe the Al\textsubscript{3}Sc phase were Rechkin et.al. in 1964 [10]. They found that the crystal structure was cubic with a lattice parameter of 4.10 Å. A few years later the correct crystallography was determined to be of the L\textsubscript{12} type, space group Pm\textsubscript{3}m [11]. This ordered structure is found in many intermetallic compounds, most renown are Cu\textsubscript{3}Au, Ni\textsubscript{3}Al and Ni\textsubscript{3}Fe. In Figure 4.4, the atomic arrangement in Al\textsubscript{3}Sc is shown. The structure appears to be face-centered cubic but in fact it is simple cubic with a base consisting of one scandium and three aluminum atoms. The lattice spacing has been most accurately determined to be 4.105 Å using X-ray diffraction [8]. According to Eq. 3.26, this leads to a lattice mismatch with the aluminum matrix of \( \delta = 0.0136 \).

In diffraction mode transmission electron microscopy, the diffraction patterns of Al\textsubscript{3}Sc precipitates are almost similar to that of fcc materials, with one significant difference. Due to the different atomic scattering factors of aluminum and scandium the structure factor (Eq. 2.4) will never be really zero. That means that the reflections that are forbidden in an fcc material (e.g. \{001\} and \{011\} reflections) will be visible in a diffraction pattern from an Al\textsubscript{3}Sc precipitate. An example is shown in Figure 4.5. It depicts the [001] diffraction pattern of a sample aged for 24 hours at 350 °C. Due to the small difference in lattice constant and the cube-on-cube...
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orientation relation, the \{200\} and \{220\} reflections of the matrix and the precipitates are not distinguishable, but the \{001\} and \{011\} superlattice reflections are a clear indication of the presence of the precipitates.

Size and shape

TEM studies of the size and shape of the precipitates are not always in good agreement with each other. An accurate study was done fairly recently by Marquis et.al. [12]. With high-resolution TEM two binary alloys were studied with different scandium content (0.1 and 0.3 wt.%). The 0.3 wt.% alloy showed diffusion-controlled coarsening of precipitates in accordance with the $t^{\frac{1}{3}}$ time dependence predicted by the Lifshitz-Slyosov-Wagner (LSW) theory. The precipitates have a facetted shape, which is established to be a regular Archimedean solid, namely a Great Rhombicuboctahedron. The alloy with 0.1 wt.% scandium shows a completely different behavior. The growth is unstable and the shapes of the precipitates show cusps and lobes and after longer aging times they become cuboidal or rod-shaped. Novotny used conventional TEM to examine a 0.2 wt.% scandium alloy [13]. Already after five hours of aging at 350 °C, a constant precipitate size is reported, which is clearly not in accordance with LSW-theory. The shape of the precipitates in this research was ‘cauliflower’-like for aging times up to 25 hours, after which a process of ‘spheroidization’ takes place. Also coexisting spherical and cuboidal particles were observed in this alloy. Signs of coherency losses were not observed for aging times shorter than 3000 hours.

There are three different TEM modes that can be used to visualize the $\text{Al}_3\text{Sc}$ precipitates: by means of strain contrast, by using the superlattice reflections or by applying phase contrast. An example of strain contrast is shown in Figure 4.6a. This sample has been aged for two hours at 400 °C. The inset shows the two beam condition used to create this bright field image. When the precipitates are small and there is still coherency with the surrounding matrix material, coherency strains can be large. Figure 4.6b is a schematic drawing of the strains around a coherent particle with a smaller lattice constant than the matrix. All the planes around the precipitate are bent and as explained in chapter 2, this will give rise to a change in contrast in a two beam bright or dark field image. The contrast is determined by the $\mathbf{g} \cdot \mathbf{R}$ product in the Howie-Whelan equations (Eqs. 2.8 and 2.9). This implies that there is a line of no contrast, where the lattice displacements are perpendicular to $\mathbf{g}$. In the case of $\text{Al}_3\text{Sc}$ precipitates in aluminum, the lattice spacing of the precipitates is actually larger than of the matrix, but the principle remains the same. Hence the
Figure 4.6 (a) Strain contrast in a sample aged for two hours at 400 °C. The typical coffee-bean contrast arises since there exists a line, over which the strain is perpendicular to \( g \) as explained in (b).

Coffee bean shaped contrast in Figure 4.6a. Strain contrast imaging is especially useful when information about the coherency and distribution of precipitates is desired.

As stated earlier, the superlattice reflections are a result of diffraction in the precipitate. When one of these spots is selected, using the objective aperture, to form the image, only electrons that have been scattered by the lattice planes of the precipitates will contribute to the image. The images 4.7a and 4.7b are made in this fashion. The sample in Figure 4.7a has been aged for two hours at 350 °C and the sample in Figure 4.7b for two hours at 400 °C. In the insets the superlattice reflection that is used to form these images has been circled. There is a significant size difference between the precipitates in both samples. One of the consequences is the much smaller intensity in the superlattice reflection as can be seen in the insets. As is the case with light diffraction from a grating, the smaller precipitates will produce less sharp diffraction spots. Even though the volume fraction of Al\(_3\)Sc is probably similar in both samples, the reflections from the larger precipitates are much more pronounced. That is why the contrast in the second picture is much better than in the first. A considerable problem, when making these kinds of images, is the long exposure time needed to get an acceptable signal to noise ratio. These two images were taken with an exposure time of one minute. When really small precipitates have to be imaged, the magnification is set high and drift becomes a serious problem. These superlattice reflection images have been used in previous investigations to estimate the size and shape of the precipitates [13]. For the larger
Figure 4.7  Two examples of superlattice reflection TEM images. (a) Aged for two hours at 350 °C. (b) Aged for two hours at 400 °C. The bigger precipitates produce a better defined superlattice reflection and hence better contrast. The particle distribution in (a) is slightly more homogeneous than in (b), where some traces of heterogeneous nucleation can be found.
precipitates such as the ones in Figure 4.7b, this is not impossible, but for the smaller particles this method becomes highly unreliable. The most accurate information about the precipitates is acquired when high resolution TEM is used. The theory behind this phase contrast imaging is described in chapter 2.

In Figure 4.8 some examples of high resolution TEM images of Al₃Sc precipitates are shown. These samples are aged at 350 °C for 1, 8, 24 and 96 hours. Especially for the smallest precipitates it is not easy to distinguish the precipitates from the

Figure 4.8 high-resolution TEM images of Al₃Sc precipitates formed at 350 °C for (a) one hour (b) eight hours (c) 24 hours and (d) 96 hours. (a), (c) and (d) are taken along a [100] zone axis; (b) along a [110] zone axis.
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Figure 4.9  MacTempas high-resolution TEM simulations of a 5x5 unit cells piece of Al$_3$Sc embedded in an aluminum matrix for varying foil thickness and defocus. For thin foils and Scherzer defocus (580 Å) the contrast is poor.

aluminum matrix. To explain the poor contrast in these images, high resolution image simulations are made using the MacTempas software package [14]. The results for a foil thickness varying between 20 and 140 Å and a defocus value varying between 0 and -720 Å is depicted in Figure 4.9. The simulated material consists of a square of 5x5 Al$_3$Sc unit cells surrounded by aluminum. The simulations are acquired by applying the multislice method as described for instance in [15]. Especially for lower thicknesses, the contrast between the precipitate and the aluminum is very poor. At Scherzer defocus (580 Å), the contrast in minimal. The smallest precipitates in this research are only a few nanometers in diameter. Furthermore, they are very likely covered on top and bottom with aluminum, which causes the contrast to be even lower than in the simulation. When the precipitates are
larger the contrast in the high resolution image will be more similar to that of the columns on the far right of Figure 4.9 and they will be more easily detected.

The high resolution images in Figure 4.8 are made with the JEOL 2010F microscope instead of with the JEOL 4000EX, which is normally the first choice for HRTEM, since it has the better point resolution. There are two reasons for choosing the 2010F. Firstly, in the 4000EX, the material did not remain stable under the electron beam. The higher energy of the 400 keV electrons induces dislocation loop formation and motion, which makes it nearly impossible to make good images. Secondly, the contrast in the 4000EX is worse than in the 2010F, caused by the smaller difference in scattering factor of scandium and aluminum atoms when the primary electron energy is higher.

A problem of the 2010F microscope is delocalization. This TEM effect is caused by the high spatial coherency of the field emission gun [16]. The high frequency oscillations in the contrast-transfer function are transferred to the final image, whereas in a microscope with a LaB₆ source they are not. As a result, a line will be imaged as a line with some weaker parallel lines nearby. In the 2010F, there will always be more or less delocalization present in the HRTEM images, which will make it more difficult to determine the exact size and shape of the precipitates.

With prolonged aging at 350 °C, the particles clearly coarsen. When aged at 400 °C, the growth is faster as can be concluded from the two precipitates in Figure 4.10. Due to the delocalization, determining the exact shape of the precipitates is difficult.

![Figure 4.10](image-url)  
*Figure 4.10  High-resolution TEM images of Al₃Sc precipitates aged at 400 °C for (a) one hour and (b) two hours.*
Figure 4.11  Image processing to determine particle size. (a) Original HRTEM image. (b) FFT of original image (c) Masking of the frequencies produced by the precipitate image (d) Inverse FFT of the masked transformation (e) Profile across the precipitate area (f) determination of the precipitate size.
They are more or less spherical but especially the larger particles (Figs. 4.8d, 4.10a and 4.10b) show some signs of faceting.

An accurate estimate of the precipitate size is important for the strengthening analysis that will follow in section 4.3. Determination of the size from the HRTEM images is however less trivial than it seems. Assuming spherical particles, their thickness and hence their visibility will decrease near the edges and consequently the measured size will be underestimated. To overcome this problem, an image processing trick is used as illustrated in Figure 4.11. In Figure (a), an HRTEM image is shown, from which the fast Fourier transform is calculated (b). This FFT has the same characteristics as the TEM diffraction pattern. The ‘superlattice reflections’ caused by the precipitates can be selected as shown in Figure (c). This masked FFT is subsequently transformed back using an inverse FFT algorithm. The result is shown in Figure (d). The precipitate stands out much more than in the original image. Its size can now be determined by plotting the intensity profile over the image as shown in Figures (e) and (f). The shape of the envelope drawn over the profile is such that it is highly unlikely that this precipitate has the Great Rhombicuboctahedron shape predicted by Marquis et. al. [12]. When viewed from the [100] direction, the central region of this Archimedean solid is of constant thickness (Fig. 4.12).

Using the method described above, the average size of the precipitates is determined for samples aged at 350 °C for times varying between 1 and 96 hours. The results are plotted in Figure 4.13. At least ten precipitates are analyzed for each aging time. The large errors indicated in the figure are firstly a consequence of the delocalization, which as explained above will cause an overestimation of the precipitate size and secondly of the fact that when looking for the precipitates in high resolution TEM, the larger particles are more likely to be found and analyzed.

Figure 4.12  Great Rhobicuboctahedron shape predicted by Wulf analysis. (a) 3D view (b) [001] projection (c) [110] projection [12].
In samples aged for very short times at a low temperature e.g. 30 minutes at 300 °C some precipitates are found, which have an unexpectedly large size (~4 nm). Evidently in the early stages of aging a broad size distribution is present, making the size determination using HRTEM nearly impossible, since the average-sized precipitates will be almost invisible.

![Figure 4.13](image)

**Figure 4.13** Precipitate sizes determined by means of the image processing technique of Figure 4.11. The samples are aged at 350 °C.

In the figure, the particle size is plotted as a function of the aging time to the power one third. Clearly the precipitate growth does not follow the $t^{\frac{1}{3}}$ relation predicted by the LSW-theory. After aging for 96 hours, the average particle size is only slightly larger than after 24 hours of aging. This arrested coarsening is one of the most useful microstructural properties of the aluminum-scandium system. Whereas the hardening phase in other heat-treatable aluminum alloys will continue to coarsen when subjected to quite moderate temperatures (150 – 200 °C) until most of the beneficial mechanical properties are lost, the Al$_3$Sc precipitates will remain small and coherent when aged at 350 °C.

Comparing our results with those of Novotny [13], some differences are observed. Where Novotny signals a constant precipitate size already after five hours of aging at 350 °C, we observe a continued coarsening up to at least 24 hours of aging. Furthermore, the observed spheroidization process as aging continues is not
observed in our experiments. Instead, the smallest particles appear spherical and the larger particles show more signs of faceting.

Coherency

Since coherency strengthening may form a major contribution to the hardness of an alloy, it is important to keep the size of the precipitates below the critical value at which the first misfit dislocation is formed to relieve the misfit strains. Approximately this will happen when the number of lattice planes at the boundary multiplied by the lattice misfit is equal to one Burgers vector. For the aluminum-Al$_3$Sc interface the critical diameter is:

\[ d_{\text{crit}} = \frac{b}{\varepsilon} = 21.0 \text{ nm} \quad (4.2) \]

However, this critical value will be higher at elevated temperatures due to the larger thermal expansion coefficient of aluminum and also due to the higher concentration of scandium in solid solution, which will slightly increase the lattice spacing of the matrix [17]. Correcting for these effects, the critical size for precipitates aged at 350 °C increases to 29 nm. This indicates that the precipitates in our research, aged at 350 °C should remain coherent for all applied aging times and indeed, no signs of coherency loss are observed in the TEM experiments.

Homogeneous / heterogeneous precipitation

In an annealed microstructure, the precipitates nucleate from the supersaturated solid solution homogeneously as can be seen in Figure 4.7. Only on grain boundaries, the precipitates will nucleate sooner and coarsen faster, due to the rapid diffusion along the boundary (See Fig. 4.14a). The shape of these larger grain boundary precipitates is not spherical but rather irregular as can be seen in the corresponding superlattice reflection image. In a deformed microstructure, the precipitation is less homogeneous. Precipitates will then nucleate preferentially on cell walls and on individual dislocations as shown in Figures 4.14b and c. These heterogeneous nucleation effects are more prominent when the aging has been performed at 400 °C as compared to aging at lower temperatures.

Superlattice reflection images are used to calculate the volume fraction of Al$_3$Sc precipitates after aging. When the size of the particles is determined using HRTEM images, the volume fraction \( f \) can be obtained by counting the number of precipitates in the image. Of course, the thickness of the foil has to be known. This can be
Figure 4.14  Heterogeneous precipitation at (a),(b) Grain boundaries (c),(d) cell strutures and (e),(f) individual dislocations. The samples are aged for (a),(b) 24 hours at 350 °C (c),(d) one hour at 400 °C and (e),(f) two hours at 400 °C.
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calculated from the electron energy loss spectrum. In an EELS spectrum a peak at zero energy loss is present caused by electrons that have not or elastically interacted with the material. Another peak is present around 50 eV energy loss arising from electrons that have lost energy by the formation of a plasmon in the specimen. This peak will be higher when the foil is thicker. From the ratio of the plasmon to zero loss peak intensity, the thickness of the foil can be determined. The volume fraction is consequently calculated as:

\[ f = \frac{n \cdot \frac{4}{3} \pi (r)^{3}}{d \cdot A} \]  

(4.3)

where \( n \) is the number of particles counted in the superlattice reflection image, \( A \) is the area of the image, \( d \) the foil thickness as determined from the EELS spectrum and \( r \) is the average precipitate radius. This analysis has been performed for samples aged for two and for 24 hours at 350 °C. For each sample five superlattice reflection images are analyzed. The calculated volume fractions are listed in Table 4.2.

<table>
<thead>
<tr>
<th>sample</th>
<th>radius r (nm)</th>
<th>volume fraction f (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 hours 350 °C</td>
<td>3.5</td>
<td>1.4±0.6</td>
</tr>
<tr>
<td>24 hours 350 °C</td>
<td>5.0</td>
<td>0.5±0.2</td>
</tr>
</tbody>
</table>

The maximum volume fraction, that will be obtained if all the scandium is in the precipitates, is equal to:

\[ f = \frac{na_{p}^{3}}{na_{p}^{3} + (100-n)a_{m}^{3}} = 0.0048, \quad n = 0.19 \cdot \frac{u_{Al}}{u_{Sc}} \cdot 4 \]  

(4.4)

In this equation 0.19 is the weight percentage of scandium in our alloy, \( u_{Al} \) and \( u_{Sc} \) are the atomic masses of aluminum and scandium. The factor 4 is present since only 25% of the atoms in a precipitate are scandium atoms and the matrix and precipitate lattice constants correct for the difference in density between the matrix and the precipitate. For the second sample, the calculated experimental value is equal to this theoretical maximum, indicating that all the scandium is in fact present in the Al1Sc phase. The experimental value obtained for the first sample is however much higher than the maximum. This can be caused by a very inhomogeneous distribution of precipitates but that is not very likely, since the five measurements are taken from very distinct regions in the specimen. A more likely explanation is an overestimation of the precipitate size. The value for the volume fraction is due to the \( r^{3} \) term very

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sensitive to the size of the precipitates. If the value is changed from 3.5 nm to 2.5 nm, a value \( f = 0.5 \) is obtained. These measurements suggest therefore, that the overestimation could be even larger than the error bars in Figure 4.13 indicate.

4.3 Dislocation mobility in an Al-0.2wt.%Sc alloy

Mechanical properties of crystalline metallic systems are almost always predominantly determined by dislocation dynamics. This section will start with a description of some of the most useful properties of aluminum-scandium alloys, namely the increased hardness and the delayed recrystallization, which are both the result of the interaction between dislocations and the Al\(_3\)Sc precipitates. An attempt is made to explain these properties by combining conventional TEM images of dislocation structures with the theory of precipitation strengthening explained in chapter 3.

4.3.1 Hardness measurements

The hardness of a material is thought to be a measure of the ease with which dislocations are nucleated and start to move. For a series of differently heat-treated Al-0.2wt.%Sc alloys, the hardness has been determined by means of three distinct methods. The first method is the classical Vickers micro-indentation technique. A square-based pyramidal indenter tip is pressed with a known load into the specimen. The size of the resulting indent is accurately determined using an optical microscope. The Vickers hardness number \( H_V \) is now the ratio between the applied load and the contact area between the indenter and the specimen.

The other two techniques used to obtain hardness values are based on nano-indentation measurements. A nano-indenter is a computer-controlled device with which a small indenter can be pressed into a material under continuous monitoring of load and displacement during both loading and unloading. The use of a nano-indenter has several advantages. The experiments are fully automated and the accuracy of the load and displacement measurements is high. No further microscopic analysis of the indents is necessary and besides a value for the hardness, the technique also provides the Young’s modulus of the material in the unloading regime.

As said, there are two ways in which the hardness can be deduced from nano-indentation. The most common technique is to use the slope of the unloading curve at the maximum indentation depth. The second technique continuously measures the hardness during loading by superimposing a small sinusoidal oscillation on the
primary loading signal. This continuous stiffness method (CSM) facilitates the continuous measurement of the material stiffness, from which the hardness can be deduced. A more detailed description of the machine and the techniques used to deduce mechanical properties from the indentation can be found in references [18,19].

In Figure 4.15 the results from the different hardness measurements are shown as a function of the aging time. The micro-indentation values are the average of five indentations per sample and the resulting hardness values are converted to SI units (MPa) by multiplying the $H_V$ numbers by 9.81. All samples are electrochemically polished before indentation to avoid any additional hardening caused by surface deformation resulting from mechanical polishing. For each heat treatment 20 nano-indentations are performed. The maximum load was set at 15 mN, which for these materials corresponds to an indentation approximately one micrometer deep. The indentations are at least 100 micrometer apart, which ensures that there is no cross-talk between the indents. The indenter used is of the Berkovich type, i.e. with a triangular pyramidal shape.

There is a large difference between the values obtained with the Vickers micro-indenter and the results obtained from the nano-indentations. The micro-indentation results are comparable with literature values [20], but the nano-indentation results

![Figure 4.15](image_url)  
*Figure 4.15  Hardness measurements plotted against aging time at 350 °C. Although the trend is similar the values obtained from nano- and micro-indentations differ considerably.*

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from both the CSM analysis and the unloading data are too high. A possible explanation is the formation of a pile-up as described in [21]. Especially in soft materials, a pile-up may form next to the indenter during loading, which leads to an underestimation of the contact area and consequently of an overestimate of the hardness. This problem is however, more prominent for the more shallow indents and should become negligibly small for deep indentations. From the CSM data the changing hardness with in increasing indentation depth can be monitored. Indeed the hardness is higher during the first part of the indentation but stabilizes after approximately 500 nm (See Figure 4.16). The values used in Figure 4.15 are an average of the values measured between 700 and 800 nanometer indentation depth i.e. after the hardness has reached a constant value.

Fortunately, the trend in the three hardness curves is identical. The increase in hardness after ten minutes of aging is large and already after 1-2 hours the peak hardness is reached.

![Figure 4.16 Hardness development during nano-indentation. The hardness values in Figure 4.15 are an average over the measured values between 700 an 800 nm indentation depth i.e. after the hardness has stabilized.](image)

4.3.2 Recrystallization effects

The increased strength is only one of the useful consequences of the formation of the Al₃Sc phase. Another aspect is the influence the precipitates have on the recrystallization behavior of the material. It is well known that randomly distributed
dispersoids hinder the motion of advancing grain boundaries. This so-called Zener drag can be expressed in the form:

\[ p_s = k \cdot f \left\langle r \right\rangle \]  

(4.5)

where \( k \) is a factor based on the interfacial energy between the dispersoid and the

Figure 4.17  OIM results showing delayed recrystallization due to \( Al_3Sc \) precipitation. An aged sample has been deformed by cold rolling and recrystallization is attempted at the indicated temperatures. Up to 550 °C no new grain nucleation occurs.
matrix. If at constant volume fraction, the precipitates coarsen, the Zener drag will decrease rapidly. This drag is such that after the aged microstructure is deformed, the temperature required for recrystallization increases. For the Al-Sc system this increase is enormous as illustrated by the measurements shown in Figure 4.17. An aged microstructure has been deformed by 70% by means of cold rolling and is subsequently annealed at the indicated temperatures. The images are orientation imaging microscopy results, which give a good impression of the state of recrystallization. Every shade of gray corresponds to a unique orientation of the crystal. An OIM image of a recrystallized microstructure shows areas with the same orientation. Clearly the presence of the precipitates has prevented the recrystallization of the heavily deformed microstructure completely up to temperatures of 550 °C. At these temperatures most of the scandium dissolves in the aluminum and the remaining precipitates will coarsen rapidly, minimizing the Zener drag on the boundaries.

The last two OIM images show the recrystallized microstructure. All scandium atoms are dissolved in the aluminum and no precipitates are present to obstruct the recrystallization front. This leads to very rapid grain formation and growth.

The Zener drag reaches its maximum for very small precipitates. Even if an annealed microstructure is deformed, after which a recrystallization is attempted, the immediate nucleation of precipitates will prevent this. This is illustrated in Figure 4.18. Here an annealed microstructure is deformed by 70% and is subsequently heated at 350 °C for two hours (Fig. 4.18a) and for 24 hours (Fig. 4.18b). The deformed microstructure consists of (screw-)dislocations, which arrange themselves in a cell structure with nearly defect free interiors and small orientation differences between the cells. This microstructure is typical for deformed aluminum specimens and is a consequence of the high stacking fault energy and hence the easy with which screw dislocations cross-slip. At the start of the heat treatment, these cells are present but the precipitates are not, but they will nucleate fast enough from the solid solution to preserve the cell structure. After annealing for 24 hours at 350 °C, the dislocations in the cell walls form a more ordered structure but the cell size remains unchanged.

The conservation of this microstructure also preserves the increased strength introduced by the cold work deformation. Hardness values for some samples that have been aged from a deformed state are given in Table 4.3. The hardness differences between the samples aged from the annealed state and those aged from the deformed state are largest after short aging times. The hardening effect of the cell structure decreases as a result of the reordering of the cell walls.
Table 4.3  Nano-indentation results for sample aged from the annealed and deformed state

<table>
<thead>
<tr>
<th>Heat treatment</th>
<th>$H_{CSM}$ (GPa)</th>
<th>$H_{Unloading}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>annealed at 640 °C</td>
<td>0.33±0.01</td>
<td>0.31±0.01</td>
</tr>
<tr>
<td>annealed + 30 mins 350 °C</td>
<td>0.83±0.03</td>
<td>0.77±0.03</td>
</tr>
<tr>
<td>annealed + 2 hrs 350 °C</td>
<td>0.87±0.02</td>
<td>0.79±0.02</td>
</tr>
<tr>
<td>annealed + 24 hrs 350 °C</td>
<td>0.81±0.02</td>
<td>0.73±0.01</td>
</tr>
<tr>
<td>70% def. + 30 mins 350 °C</td>
<td>0.99±0.04</td>
<td>0.91±0.03</td>
</tr>
<tr>
<td>70% def. + 2 hrs 350 °C</td>
<td>1.08±0.05</td>
<td>1.01±0.04</td>
</tr>
<tr>
<td>70% def. + 24 hrs 350 °C</td>
<td>0.88±0.09</td>
<td>0.80±0.09</td>
</tr>
</tbody>
</table>

4.3.3 Strengthening analysis

In this section the theory of precipitation hardening as described in Chapter 3 will be applied to the Al-Sc alloy. This analysis follows closely the approach of Seidman et.al. [22], who analyzed an Al-0.3wt.%Sc alloy and compared its hardness to calculated values for the different contributions of precipitation hardening. Since stacking fault strengthening is not considered to play a major role in these alloys, due to the high stacking fault energy of aluminum and the small size of the precipitates, we will focus our analysis on coherency hardening, modulus hardening and order strengthening. In Figure 4.19, the different strengthening contributions as explained in the following text are plotted as a function of the precipitate radius. The material constants used are given in Table 4.4.

![Figure 4.18](image)

Figure 4.18  After deformation of an annealed microstructure, aging at 350 °C does not lead to recrystallization but to a stabilization of the cell structure. (a) After two hours aging the cell walls are formed by many dislocations which (b) after 24 hours have partly annihilated and partly rearranged to form a more ordered, recovered microstructure. The cell size however remains unchanged.
Figure 4.19 Contributions of the different strengthening mechanisms plotted against the precipitate size. In the underaging regime, the sum of coherency ($\tau_{cC}$) and modulus ($\tau_{c\mu}$) strengthening or order strengthening ($\tau_{cO}$) determines the yield stress, depending on which has the highest contribution.

Coherency hardening

The theory derived in Chapter 3 leading to Eq. 3.24 is in fact an analysis of the elastic interactions between the stress fields present around a coherent spherical particle and a straight edge dislocation. The assumption that the precipitates are spherical is justified in our material since the HRTEM images show little evidence of other shapes. The fact that the analysis only takes into account straight edge dislocations, is questionable. For an edge dislocation the interaction is maximal. For many systems, including aluminum alloys, the flow stress is however considered to be mainly governed by screw dislocations. A pure screw dislocation in an isotropic medium has no elastic coupling with the coherency stress fields. Furthermore, when an edge dislocation interacts strongly with a precipitate it will never remain straight and more screw parts will arise. All this leads to the conclusion that Eq. 3.24 strongly overestimates the actual critical resolved shear stress due to coherency strains.
When the precipitates are strong and the break away angle consequently becomes small, Eq. 3.16 has to be used instead of Eq. 3.15. This can be used to calculate the maximum contribution of coherency hardening before the Orowan bypassing mechanism takes over. According to Brown and Ham [23], still under the straight edge assumption, this maximum stress is given by:

$$\tau_{c,\text{max}} = 1.84 \mu \varepsilon f^{3/4}$$  

This maximal value is also taken into account in Figure 4.19. The meaning of the different variables in this and following equations is explained in Chapter 3 and can also be obtained from Table 4.4.

**Modulus hardening**

In Figure 4.19 the increase in critical resolved shear stress as a consequence of the modulus mismatch between the matrix and precipitates is plotted. The values are obtained by applying Eq. 3.23.

**Order strengthening**

The Al$_3$Sc precipitates are of the L1$_2$ ordered crystal structure. This implies that when a dislocation passes through the material and displaces the atoms above a {111} plane by a Burgers vector with respect to the underlying material, the resulting stacking is not perfect anymore. In fact, an anti-phase boundary (APB) is created. The energy associated with this planar defect is hard to establish. According to [3], reported values vary between 0.3 and 0.7 J/m$^2$.

For weak obstacles the increase of the critical resolved shear stress is given by Eq. 3.28. This equation does not consider the possibility of a second dislocation, which would restore order in the precipitate. The presence of such a dislocation would lower the critical stress significantly. This is expressed in Eq. 3.31. However, in our material the volume fraction Al$_3$Sc is very low and the distance between the two paired dislocations can be calculated to be 32 nm, which is larger than in the systems for which such coupled dislocations are actually observed. In the TEM investigation in section 4.3.4, no coupled dislocations are observed and the application of Eq. 3.31 therefore likely underestimates the hardening effect of the APB formation in these alloys.

For strong obstacles close to the Orowan limit, substitution of Eq. 3.27 in Eq. 3.6 and Eq. 3.16 and further using Eq. 3.18 leads to a maximum value for $\tau_{cO}$ of:
Again, this is without considering the possibility of a trailing dislocation. The counterpart of Eq. 3.31 for strong obstacles is:

\[ \tau_{\text{r},\text{max}} = \frac{0.8\gamma_{\text{APB}}}{b} \left( \frac{3\pi f}{8} \right)^{\frac{1}{2}} \]  

(4.7)

In Figure 4.19, Eq. 3.28 and Eq. 3.31 are plotted until they reach the maximum value predicted by Eqs. 4.7 and 4.8. In reality the transition is of course more gradual than shown here.

Since both modulus and coherency strengthening are at a maximum when the dislocation is just about to enter the precipitate, these contributions can be added as is done in Figure 4.19. The order strengthening term \( \tau_{\text{r},\text{max}} \) however, reaches a maximum when the dislocation is exactly halfway the precipitate, i.e. when the largest amount of new APB surface has to be created. The yield strength of the material is now determined by the highest of the two contributions namely order strengthening and the sum of coherency and modulus hardening.

Looking at the different curves in Figure 4.19, it can be concluded that coherency strengthening is indeed the least important contributor to the hardness. In addition, this value is, as explained above, most likely overestimated. The difference between the two order strengthening curves is large. If we assume that the dislocations travel in pairs, then in the underaging regime the hardness is almost entirely determined by modulus hardening. This leads to an increase in critical resolved shear stress of around 75 MPa at peak hardness, which occurs at a precipitate radius of 3.0 nm. If however the assumption is made, the dislocation do not pair up and every dislocation can be treated as existing solitary, the order contribution suddenly is the major hardening contributor, increasing the critical resolved shear stress by no less than 106 MPa until at a critical radius of 2.3 nm and overaging sets in and Orowan looping takes over.

To compare these theoretical results to our hardness measurements we have to correct for the fact that our indents are made on arbitrary grains in a polycrystalline material. The hardness is determined as in an uniaxial compression test, i.e. equal to the average yield stress. For a polycrystalline material, exhibiting Taylor-like behavior, the shear stress is approximately equal to one third of the hardness. From Figure 4.15, the difference between peak hardness and the hardness before aging is 0.42 GPa for the micro indentations, 0.48 GPa for the unloading curve results from
nano-indentations and 0.53 GPa from the CSM measurements. If only the ordering contribution to the strength is considered, the maximum increase in shear stress is according to Figure 4.19 equal to 106 MPa. Multiplied by three this leads to values comparable to the hardness measurements.

<table>
<thead>
<tr>
<th>Constant</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shear modulus $\mu$</td>
<td>25 GPa [24]</td>
</tr>
<tr>
<td>modulus mismatch $\Delta \mu$</td>
<td>43 GPa [25]</td>
</tr>
<tr>
<td>Coherency pre-factor $\chi$</td>
<td>2.6 [26]</td>
</tr>
<tr>
<td>Burgers vector $b$</td>
<td>0.286 nm</td>
</tr>
<tr>
<td>Constrained lattice mismatch $\varepsilon$</td>
<td>9.07·10^{-3}</td>
</tr>
<tr>
<td>Poisson’s ratio $\nu$</td>
<td>0.34</td>
</tr>
<tr>
<td>Anti-phase boundary energy $\gamma_{ABP}$</td>
<td>0.5 J/m² [3]</td>
</tr>
<tr>
<td>Volume fraction $f$</td>
<td>0.0048</td>
</tr>
</tbody>
</table>

4.3.4 Observation of dislocations

To study the dislocation mobility on the scale of the precipitates, a large number of TEM specimens are made. Most of them are deformed ex-situ. A TEM sample is cut out of a macroscopically deformed piece of material. Another option is to deform the specimens in-situ in the TEM. For this purpose a single-tilt straining holder is used. Both methods have their specific advantages and disadvantages. The ex-situ deformed samples will contain a homogeneous distribution of dislocations and is well suited to look for signs of Orowan by-passing. The observed dislocation structure is however a relaxed one. Not only is the sample not loaded during observation, but also during the preparation stages some degree of relaxation may take place.

The samples that are deformed in-situ will show the dislocations under an applied load, but the situation in the thin foil is not the same as in a bulk specimen. Only when the slip plane is more or less parallel to the foil, a good observation of moving dislocations can be made. Another disadvantage of the in-situ technique is that the deformation will be concentrated in the weakest part of the specimen, which may for instance be a slight tear in the edge of the hole. Furthermore, when deformation is induced inside a thin foil, it will have a tendency to buckle. This will cause orientation gradients over the field of view in the microscope, which will decrease the quality of the image.
Figure 4.20 (a) Weak beam TEM image of dislocations in an ex-situ deformed sample aged for 1 hour at 400 °C. In this overaged state many small loops can be seen, indicative of the Orowan bypass mechanism. (b) Enlarged view of framed section.

A good alternative is to take the best of both techniques i.e. with ex-situ deformation a high homogeneous dislocation density is introduced after which TEM samples suitable for in-situ deformation are prepared. By subsequently applying loads in-situ, the already present dislocations may be observed under tension.

Figure 4.20 and 4.21 show weak beam TEM results obtained with the 4000EX microscope. The samples are ex-situ deformed by tensile straining and subsequently prepared for TEM. The sample in Figure 4.20 is aged for one hour at 400 °C, which leads to precipitates with an average diameter of around 15 nm. According to Figure 4.19 this is clearly in the Orowan regime. Indeed many dislocation loops are observed as is visible in the enlarged section of Figure 4.20. The loops are examined using the invisibility criterion explained in Chapter 2. The Burgers vector of these dislocations is of the type $b = \frac{1}{2}a <110>$. This excludes the possibility of the dislocations being quenched-in Frank loops, since these dislocations would possess a Burgers vector $b = \frac{1}{2}a <111>$.

Figure 4.21 shows similar results but now for an alloy aged for only 30 minutes at 300 °C, so a sample which is without doubt in the underaged state. Theory predicts pairs of dislocations shearing through the ordered precipitates. Except for the pair marked by the arrow, no coupling of dislocations is observed. Some loops however are visible. Since it is highly unlikely that the average-sized precipitates in this sample, which are not expected to be larger than one nanometer, are bypassed by the
Figure 4.21  (a) Weak beam TEM image of dislocations in an underaged sample (aged for 30 minutes at 300 °C). The arrow indicates a possible pairing of dislocations but this is not observed regularly. (b) Enlarged view of framed section showing dislocation loops.

Orowan mechanism, we conclude that the loops are caused by the large size distribution present in the underaged samples. The density of loops is much lower than in the overaged sample.

An example of an in-situ deformation experiment is shown in Figure 4.22. In this experiment a sample aged for 30 minutes at 350 °C is inserted into the microscope nearly defect-free. On straining this sample, several slip planes are activated, two of which are shown in Figure (a). The contrast in this picture is caused by dislocation activity on two planes intersecting the specimen foil at an inclined angle with respect to the viewing direction. Outside these planes no dislocation activity occurs. In the weak beam image (Fig. 4.22b), one of the slip planes is shown. The dislocations traversing this plane are bent through large angles around the precipitates as seen in the regions indicated by arrows. This implies that the precipitates in this sample, which is according to Figure 4.15 in an underaged state, can be considered as strong obstacles for dislocation motion. After breaking free of the particles, the dislocations leave a contrast at the precipitates, which is not seen outside the slip planes. This band of contrast is not caused by bending of the foil, since its location is not influenced by a slight tilt of the specimen. The most logical explanation is the formation of Orowan loops when the dislocations pass the precipitates. Again since this is an underaged specimen, only a fraction of the precipitates is assumed not to
be sheared. This fraction is however considerable as can be concluded from these images.

It is concluded that in an overaged specimen the dislocations pass the precipitates by use of the Orowan looping mechanism. In the underaged specimens the size distribution is large and some precipitates are too strong to be sheared, resulting in dislocation loops. Hardness measurements indicate however, that shearing must be dominant, since for these heat treatments the hardness has not reached its maximum value. No paired dislocations are observed. This implies that the corrections to the contribution of order strengthening caused by the second dislocation, which lower the critical resolved shear stress, should not be applied and Eqs. 3.28 and 4.7 are to be used. This makes order strengthening the dominant hardening mechanism in underaged alloys.

Figure 4.22  (a) Bright field TEM image taken during an in-situ deformation experiment of a sample aged for 30 minutes at 350 °C. Most dislocation activity is confined to two slip planes, which intersect the foil at an inclined angle with respect to the sample surface. (b) Weak beam image of one of the slip planes. The arrows indicate dislocations which are bent through large angles around the precipitates. This indicates the strength of the precipitates in this underaged state. Upon traversing the plane, dislocations leave contrast at the precipitates most likely a consequence of Orowan bypassing.

4.4 Addition of scandium to other alloys

Aluminum alloys are generally categorized in two classes: heat-treatable and non-heat-treatable alloys. Following the international AA designation, the heat treatable alloys are the 2xxx series (Al-Cu alloys), the 6xxx series (Al-Mg-Si) and the 7xxx series (Al-Zn-Mg). These are the alloys get their strength from artificial aging procedures and hence from precipitation hardening. At first, addition of scandium to
one of the heat-treatable alloys in order to benefit from the added effect of the Al$_3$Sc-induced hardening, seems the most obvious. However, the temperatures at which these alloys are normally heat-treated differ significantly from the high temperatures, which are as explained before, necessary to heat-treat the Al-Sc system. For instance the aging temperatures for 2xxx alloys vary between 160 and 190 °C, for 6xxx alloys between 160 and 205 °C and for 7xxx alloys between 95 and 180 °C [27]. All these temperatures are considerably lower than 350 °C, at which the Al$_3$Sc phase in this research is formed.

Adding scandium to non-heat-treatable alloys as for instance the 1xxx series (>99% pure Al), the 3xxx series (Al-Mn) and the 5xxx (Al-Mg) is however promising. These alloys get their strength from strain hardening and from solid solution strengthening. The 5xxx alloys are characterized by their good corrosion resistance, workability and weldability but they have a reduced strength and a low recrystallization temperature, which results in large grain sizes when used in the cast or recrystallized state [28]. The complementary qualities of the addition of scandium are obvious.

For this research an alloy has been made, which has the composition shown in Table 4.5.

<table>
<thead>
<tr>
<th>element</th>
<th>Al</th>
<th>Sc</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>other</th>
</tr>
</thead>
<tbody>
<tr>
<td>concen. (wt.%)</td>
<td>97.6</td>
<td>0.19</td>
<td>2.1</td>
<td>0.04</td>
<td>0.07</td>
<td>&lt;0.005</td>
</tr>
</tbody>
</table>

The solubility of magnesium in aluminum is high with a maximum at 450 °C. At this temperature 17.4 wt.% magnesium dissolves [24]. Most 5xxx alloys therefore contain more than 2 wt.% magnesium. In combination with scandium however, some care has to be taken at the solution temperature. To dissolve 0.19 wt.% scandium in aluminum, the temperature has to be raised to at least 600 °C. This is however also the temperature above which an Al-2.0wt.%Mg alloys starts to melt. There is therefore only a narrow temperature regime, in which both the magnesium and the scandium form a solid solution with the aluminum. For this reason a lower solution heat treatment temperature of 600 °C is used for these alloys than for the binary Al-Sc material.

In Figure 4.23 the results of hardness measurements are shown obtained with the CSM method from nano-indentations. The samples in these measurements have been solution treated at 600 °C for 30 minutes and water quenched. Subsequently, they are aged at 350 °C during periods varying between 10 minutes and 96 hours. Contrary to the binary alloys these samples are not electrochemically polished, since
magnesium tends to diffuse out of the material layer and form a magnesium oxide layer at the surface. Instead, they are mechanically polished to a mirror-like finish using a SiO$_2$ colloidal suspension. This might leave a thin deformed layer at the surface. However, since the observed hardness has attained a constant value with increasing indentation depth, it is believed that no significant hardening from the polishing is present in our results. The results for the binary Al-Sc alloy are also plotted for comparison. The hardness increase for the annealed specimens is enormous. The ternary alloy in this state is more than 300 MPa harder than the binary alloy. This difference, however, depends on the aging time and at peak strength is only 100 MPa. The shape of the hardness curve for the ternary alloy is slightly flatter than for the binary alloy, which can be attributed to a more dominant role of the size independent order strengthening. According to [20], the lattice mismatch between matrix and precipitates is decreased by the addition of 2 wt.% magnesium from 0.1365 to 0.111. This will decrease the hardening effect due to coherency strengthening.
4.5 Conclusions

Scandium additions to aluminum alloys are promising. Due to the homogeneous distribution of nano-sized Al₃Sc precipitates high strengths can be achieved. The formation of the precipitates increases the hardness by several hundreds MPa. Furthermore, these precipitates exert a strong Zener drag on advancing dislocations and grain boundaries, which hinders recrystallization and thereby facilitates the formation of small grained materials.

These alloys differentiate themselves from other aluminum alloys by their excellent properties at elevated temperatures. The diffusion of scandium in aluminum is slow and consequently the precipitates coarsen slowly. After 24 hours of aging at 350 °C, the coarsening has stopped almost completely. This implies that contrary to other aluminum alloys the mechanical properties at temperatures up to 350 °C can be preserved. Since also the cell or grain structure remains largely unaltered, the entire microstructure is stable at these temperatures.

The strengthening qualities of the Al₃Sc phase can be attributed to modulus hardening, coherency hardening and order strengthening. In-situ TEM results indicate no coupling of dislocations in underaged specimen. This increases the contribution of order strengthening by more than a factor two, making it the dominant strengthening effect in this material.

Scandium can be added to other aluminum alloys, but this often gives rise to difficulties concerning heat treatments. Adding scandium to aluminum magnesium alloys can be very useful. The precipitation strengthening and grain refining qualities of scandium are complementary to the mechanical properties of the aluminum magnesium alloy.
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References

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4. Binary Alloy Phase Diagrams