Mapping the microstructure of a friction-stir welded (FSW) Al-Li-Cu alloy

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Abstract. The friction-stir welding (FSW) process induces both heat and deformation which lead to inhomogeneous precipitation microstructures in structural hardening alloys. A map of this microstructure can be obtained by 2D scanning SAXS in alloys where (a) a single phase is precipitating and (b) the precipitates are roughly isotropic. In Al-Li-Cu alloys, these conditions are not fulfilled. Very anisotropic precipitates are forming (T₁, θ’) with aspect ratios in the range 10 to 100. The inhomogeneity in the texture of the material (due to the deformation and recrystallisation) is a strong obstacle to the interpretation of the SAXS signal. This paper is an attempt to apply systematic simple interpretation models to characterise the precipitation microstructure across the weld area. It shows that in certain condition, it is possible to apply simple Guinier-type plot to extract the morphologies (length and thickness) of the particles.

1. Introduction
The third generation of Al-Li alloys are subject to many studies due to the urge for weight reduction in aerospace industry. Further weight savings can be obtained by the use of friction-stir welding (FSW) instead of riveting. The FSW process is a solid state welding technique that induces both heat and deformation. Depending on the way they have been affected by the weld, four zones can be defined in the material:

- the unaffected base material (BM)
- the heat affected zone (HAZ)
- the thermo-mechanically affected zone (TMAZ)
- the weld nugget (WN) at the centre of the weld, where recrystallisation has occurred

This leads to a complex precipitate microstructure which creates a gradient of mechanical and corrosion properties. Microstructure mapping of FSW welds has been successfully obtained by small angle X-ray scattering (SAXS) before [1], but in the case of Al-Li-Cu alloys, there are two obstacles to the interpretation of the SAXS profiles. First, at least two phases are likely to form (T₁, Al₂CuLi forming platelets in the 111 planes of aluminium and θ’, Al₂Cu, forming platelets in the 001 planes of aluminium). Other phases might form as well (e.g. δ’, Al₃Li) but they will not be considered in this study. Then, the strong anisotropy of the T₁ and θ’ precipitates might cause misinterpretation of the data, as the texture of the material is affected by the FSW process. This preliminary study aims at assessing the use of SAXS as a tool for mapping the microstructure of Al-Li-Cu alloys in the different zones of the weld.
Table 1. Composition of the Al-Li-Cu alloys (weight %).

| Alloy | Cu   | Li    | Mg    | Ag    | Zr     |
|-------|------|-------|-------|-------|--------|
| 2098  | 3.2-3.8 | 0.8-1.3 | 0.25-0.8 | 0.25-0.6 | 0.04-0.18 |
| 2050  | 3.2-3.9 | 0.7-1.3 | 0.2-0.6  | 0.2-0.7  | 0.06-0.14 |

2. Experimental procedure

2.1. Materials

Two types of alloys have been used in this study. The first is a 2098 alloy that has been studied in another work [2] and the other is a 2050. These two alloys have very similar composition, as can be seen in Table 1, and were provided by Alcan. Friction stir welding was performed by Onera for the 2098 and by Eads-IW in the framework of the Coralis project (see acknowledgments) for the 2050. For 2098, FSW was performed in T8 condition. One of the 2098 alloy was then heat treated at 100°C for 1000h to artificially age the alloy. For the 2050 alloy, FSW was performed in T3 condition and then aged to T8 condition.

Two types of precipitates are expected to form in the alloy [3]:

- $T_1$ (Al$_2$CuLi): platelets laying in the (111) planes of the aluminium matrix.
- $\theta'$ (Al$_2$Cu): platelets laying in the (001) planes of the aluminium matrix.

Other phases such as $\delta'$ (Al$_3$Li) or S (Al$_2$CuMg) might form as well but are considered in the present study as negligible (this is justified by TEM experiments, not shown here). Other Cu-rich objects are likely to precipitate: Guinier-Preston (GP) zones and/or $\theta''$. As their habit planes are the aluminium (002), they are difficult to separate from the $\theta'$ precipitates. As this distinction is not crucial, we will consider all these objects as $\theta'$-like particles.

2.2. SAXS experiments and interpretation

The small-angle scattering experiments have been performed at the BM02-D2AM beamline at the European synchrotron radiation facility (ESRF). The samples were cut perpendicularly to the weld line and were mechanically polished down to a thickness of about 100µm. The energy was set to 8.3keV and the beam size was about 200µm x 200µm. The sample to detector distance was 33.6cm (as determined by silver behenate diffraction).

We have performed 1D scans across the FSW welds, recording a SAXS image every 200µm. For the 2050 sample, we have performed a 2D scan with a 500µm step to map the whole weld region.

Typical SAXS images collected on the CCD detector can be seen on figure 1 and 2. These images have been extracted from the 1D scans across the weld line. The images present streaking that is typical from anisotropic precipitates in a textured alloy. Except in the base material, the grain texture of the alloy is unknown and varies a lot across the weld, from the recrystallised weld nugget to the thermo-mechanically affected zone.

The anisotropic nature of the SAXS intensity could in principle be accounted for by using intensity sectors instead of azimuthally averaged intensity, or even by fitting directly the image by a 2D intensity model. This is not practical in this particular case for two reasons. First, the actual texture of the material, and more specifically the out of plane texture, is unknown (and varies across the welding plane), so that modelling of the data is difficult. Second, 2D and 1D scans lead to a lot of SAXS images and analysing manually selected intensity sectors would be extremely time-consuming. We use azimuthally averaged images to process the SAXS images, and extract data for platelets particles, bearing in mind the following considerations:
• The grain texture of the material is not random and not homogeneous.
• Two types of precipitates (T\textsubscript{1} and $\theta'$) with different electronic contrast are present in the material in such a way that they are indistinguishable.

If the distribution of orientations around the average texture of the material is broad enough, the averaged SAXS intensity should be characteristic of the morphologies of the precipitates and this should be true no matter the orientation of the sample.

Figure 3(a) shows such an azimuthally averaged SAXS image (double log plot). The high q region follows a classic $q^{-4}$ Porod law, whereas the intermediate q region follows a $q^{-2}$ behaviour, typical from a flat specimen. This behaviour (true in every zone of the weld) justifies the use of the azimuthally averaged intensity. We can then assume that the intensity is of the form (cf. [4], pp 17-51):

$$I(q) = A \frac{2\pi}{q^2} (\Delta \rho)^2 t^2 \exp\left(-\frac{q^2 t^2}{12}\right)$$

where $A$ is the area of the particles, $\Delta \rho$, their electronic contrast with the aluminium matrix and $t$ is their thickness. A Guinier-type plot Log$(Iq^2)$ vs. $q^2$ can then be used to extract the thickness $t$ of the precipitates. Figure 3(b) is an example of such a plot. It clearly shows a linear part that can be used to fit a Guinier law to extract the thickness.

In principle, the relation (1) and its associated Guinier plot can also be used to extract the length of the particles via their cross-section area A. However, although the q=0 intensity of this Guinier plot is proportional to $A$, it also reflects the texture of the material. This non-uniform texture will not alter the slope of the Guinier plot, but is likely to modify the q=0 intensity.
Figure 3. Azimuthally averaged SAXS image extracted from a 1D scan across the weld line for the 2098 alloy welded in T8 condition. (a) Double-log plot. The $q^{-2}$ region before the classic $q^{-4}$ Porod law is typical from flat precipitates. (b) Guinier-type plot $\log(Iq^2)$ vs. $q^2$. The linear part gives the thickness according to eq. (1).

To obtain the length of the particles, we have performed a classic Guinier plot $\log(I)$ vs. $q^2$ in the very low $q$ range. Although this is far from being a perfect solution, the initial slope of the Guinier plot should still give the correct tendency for the evolution of the particles length, even if the absolute scale is incorrect.

3. Results and discussion

3.1. Temporal stability of the welded 2098 alloy

Although the 2098 alloy is considered to be stable in T8 condition, the temporal stability of the alloy will be modified by the FSW process. To analyse this modification, an artificially accelerated ageing treatment has been applied (1000h at 100°C) to the 2098 alloy. Figure 4 shows the morphologies obtained from the Guinier plots for the 2098 alloys welded in T8 condition with and without artificial ageing.

Figure 4(a) seems to indicate that little variation in morphology is observed across the weld. The thickness is constant and the length is slightly lower at the centre of the weld. This is slightly misleading in the sense that two important piece of information are missing: the volume fraction and the type of precipitates ($T_1$ or $\theta'$). Although we know that mainly $T_1$ is present in the base material [2], it is very likely that $\theta'$-type precipitates are forming in the weld zone. Denquin et al [2] have observed only sparse particles in the weld, suggesting a dissolution of the $T_1$ precipitates. Our results suggest that, if this is the case, the remaining $T_1$ or the $\theta'$-type precipitates which may have formed are of similar size.

Figure 4(b) shows the morphologies obtained for the 2098 alloy artificially aged 1000h at 100°C. In contrast to what is observed in the non-aged alloys, the 4 FSW zones (BM, HAZ, TMAZ and WN) can be distinguished, and are labeled on the figure. The increase in thickness in the HAZ zone can be interpreted as a thickening of $T_1$ precipitates and is consistent with figure 2(b) which shows similar patterns than figure 2(a), but in a smaller $q$-range. In the TMAZ and WN zones, the SAXS patterns clearly change (cf. figures 2(c) and 2(d)) and the morphologies measured in figure 4(b) are most likely due to $\theta'$-type precipitates.

From the 1D scans obtained on the 2098 alloy welded in T8 condition with and without
ageing, it can be concluded that during an artificial ageing, $\theta'$-type precipitates are forming in the TMAZ and WN zones with the solute content issued from the dissolution of the initial $T_1$ platelets. In the HAZ, the results may be interpreted as a thickening of remaining $T_1$ platelets.

3.2. 2D microstructure mapping on the 2050 alloy welded in the T8 condition

To further probe the anisotropy of the microstructure, it might be necessary to use 2D scanning SAXS mapping of the FSW weld. This has been attempted on a 2050 Al-Li-Cu alloy welded in the T8 condition. The data has been processed in the same way than for the 1D scans. The results are plotted in figure 5 for the precipitates thicknesses and figure 6 for the lengths.

There is little evolution in terms of precipitates thickness, as in the case of the 2098 alloy welded in T8 condition. In contrast, however, the precipitates are slightly shorter in the weld.
As already discussed, the inhomogeneities in the texture of the material do not permit the use of the integrated intensity as a tool for probing the volume fraction, as the intensity in the plane of the detector is very dependent on the actual texture of the irradiated material. However, if we consider that the Fourier transform of a flat precipitate is a streak in the direction normal to the habit plane of the precipitate, and we consider the fact that this streak will give intensity at large q only when it is oriented not too far from the plane of the detector, it can be expected that the intensity at very low angle will be less influenced by the texture and will better reflect the amount of scattering matter.

With this in mind, we have chosen to use the extrapolated intensity at q=0 as an estimation of the relative volume fraction in the material. Figure 7 shows the evolution map of I(q=0) in the weld area. If I(q=0) is indeed a good indication of the behaviour of the volume fraction, figure 7 seems to show a much lower volume fraction in the weld nugget than in the base material. There is also the indication of an increase of the volume fraction in a zone that is likely to be the TMAZ. These observations are consistent with the fact that T1 phase nucleation is very sensitive to the presence of dislocations [5]. As the T8 precipitation heat treatment has been applied after the FSW welding process, the low amount of dislocations in the recrystallised nugget is less favourable to T1 nucleation whereas the TMAZ zone has undergone a further deformation during the weld, with a likely increase in dislocation density, which is in favour of a higher T1 number density.

4. Conclusion
This preliminary study has shown that scanning SAXS can be used to map the microstructure of a FSW aluminium alloy, even in the case of anisotropic precipitates. However, care must be taken in the interpretation of the results in terms of precipitates volume fraction because of the variation of the material texture due to the deformation induced by the welding process and the recrystallisation that occurs in the weld nugget. The presence of different phases, which is the norm rather than the exception in aluminium alloys, should also be considered carefully, as it is impossible to ascertain which phase is present at which data point. To overcome these limitations, scanning SAXS should then be used in combination with other characterisation techniques such as transmission electron microscopy (TEM) for determining the phase forming in each zones, electron back scattered diffraction (EBSD) to map the texture in the weld area and/or differential scanning calorimetry (DSC) to help with the calibration of the precipitates volume fraction.

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