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DOI 10.1088/1742-6596/262/1/012052

**Publication date** 2010 **Document Version** Final published version

Published in 12th International Workshop on Slow Positron Beam Techniques SLOPOS 12

### Citation (APA)

Ruiz Ripoll, ML., Schut, H., Alderliesten, RC., van der Zwaag, S., & Benedictus, R. (2010). The generation of deformation damage during fatigue loading in Al-Cu alloy studied by the Doppler Broadening technique. In S. Buckman (Ed.), 12th International Workshop on Slow Positron Beam Techniques SLOPOS 12 (pp. 1-4). (Journal of Physics Conference Series; Vol. 262 (2011)). ARC Centre of Excellence for Antimatter-Matter Studies. https://doi.org/10.1088/1742-6596/262/1/012052

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To cite this article: M L Ruiz-Ripoll et al 2011 J. Phys.: Conf. Ser. 262 012052

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### The generation of deformation damage during fatigue loading in Al-Cu alloy studied by the Doppler Broadening technique

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**Abstract**. We have investigated the evolution of damage during fatigue loading in Al–Cu 2024-T3 alloys using the positron annihilation Doppler Broadening (DB) technique. This technique enables us to monitor in a non destructive way, at the atomic and vacancy level, the formation of deformation defects and their interaction with solute atoms at selected stages of fatigue testing. The changes in the S and W Doppler Broadening parameters are linked to the changes in fatigue behavior at lower stress levels. The material was tested under constant amplitude fatigue loading at four different stress levels and DB tests were conducted at selected stages of fatigue lifetime. The results are compared to those obtained during static tensile tests.

### 1. Introduction

Plastic deformation of precipitation hardened aluminium alloys leads to the displacement of dislocations through the matrix containing a dense network of precipitates. With increasing deformation levels the defects link to form voids which initiate nano sized cracks that subsequently grow and ultimately lead to failure of the material [1-2]. In order to observe the evolution of these defects and to monitor the damage process directly, the positron annihilation Doppler Broadening (DB) technique has been used. Positrons are effectively trapped at defects associated with open volume defects such as vacancies, vacancy clusters and misfit location at the precipitate/matrix interface or inside solute aggregates with higher positron affinity than the host material. Upon trapping a positron annihilates with an electron into two 511 keV photons. Because of the momentum of the electron the measured energy of the photon is shifted by an amount of  $\Delta E = p_1 c/2$ , with c the speed of light and p<sub>L</sub> the longitudinal component of the positron-electron momentum along the direction of emission of the photons. In an annihilation spectrum these shifts lead to a broadening of the 511 keV photo peak which is quantified by the S- and W-parameters [1]. S and W reflect annihilations with valence and core electrons, respectively, and are therefore ideal to monitor the generation and evolution of deformation induced defects and the chemical environment at the positron trapping sites. We have used these properties to investigate the role of Cu solute atoms on the evolution of damage and the effect of intermittent annealing periods on the fatigue lifetime [2-6].

### 2. Experimental

Commercial aluminium 2024-T3 (AlCu4Mg1) sheet metal has been used as base material. The material has been solution treated at 495 °C then cold worked and finally aged at RT. The useful length of the dog-bone shaped specimens was 120 mm with 1.2 mm thickness. The total length was 240 mm and the radius of transition was 185 mm ( $k_{i}$ =1.1). The Al alloy contains Cu (3.80-4.90 at.%), Mg (1.20-1.80 at.%), Mn (0.30-0.90 at.%), Fe (0.50 at.%), Si (0.50 at.%), Zn (0.25 at.%) and other constituents with concentrations below 0.15 at.%.

Two different routes for mechanical loading of the samples have been followed. The first one consists of continuous constant amplitude fatigue tests at selected stress levels until failure (referred to as T3-C). The second route combines fatigue testing interrupted by annealing periods (referred to as T3-A). The T3-A sequence comprises of:

- 1. Fatigue loading for 50, 100, 200 and 400 keycles depending on the maximum stress  $\sigma_m$  applied. These were  $\sigma_1=0.97$ ,  $\sigma_2=0.89$ ,  $\sigma_3=0.81$  and  $\sigma_4=0.76$  of the yield strength of 345 MPa [7]. The stress ratio (R =  $\sigma_{min}/\sigma_{max}$ ) was in all cases 0.1.
- 2. A Doppler Broadening measurement to monitor the effect of the mechanical loading.
- 3. An annealing period during which the material is annealed at 65 °C for 24h (in air) and thereafter quenched in water at room temperature.
- 4. A second DB measurement to investigate the effect of the annealing during step 3.
- 5. Repetition of the above steps until failure.

The fatigue tests were conducted using a servo hydraulic testing machine with a 60 kN load capacity and a steel alloy grip based on ASTM E606 [8] at 25 Hz frequency.

The original condition of each sample was established before the loading with an additional DB test. To avoid further natural aging effects the specimens were stored at -20 °C after each fatigue sequence and DB test. The DB experiments were carried out using a <sup>22</sup>Na source sealed between two 7 µm thick Kapton foils, sandwiched between two identical specimens. Two HPGe detectors were used to detect both annihilation photons in time coincidence with an overall energy resolution of 0.9 keV at 511 keV. The S parameter is calculated as the ratio of the counts registered in a fixed central momentum window ( $|p_{l/l}| < 3.5 \times 10^{-3} \text{ m}_{o}\text{c}$ ) to the total number of counts in the photo peak. This choice of the momentum window makes the S parameter sensitive to annihilations with low momentum valence electrons. Similarly, the W parameter is obtained from the high momentum regions (Wleft and  $W_{\text{right}}$  with  $10 \times 10^{-3} m_0 c < |p_{//}| < 26 \times 10^{-3} m_0 c$ ) and accounts for annihilations with high momentum core electrons. All S and W data presented in this paper are normalized to the S and W values of pure, well annealed aluminium.

### 3. Results

In figure 1 the number of cycles to failure as a function of the maximum load ( $\sigma_m$ ) is shown in an S-N plot. It can be seen that the results follow the trend of a typical Wöhler curve. In the case where fatigue and annealing periods are alternated (T3-A), unlike the results presented by Lumley [2] no improvements in life times were observed with respect to those for the continuously loaded sample (T3-C), but the amount of scatter in lifetime seemed to be smaller, in agreement with his earlier observations. In figure 2 the Doppler-broadening data are shown together with those of the as prepared and the failed T3-C material. Also shown are the lines connecting the S,W points for pure Al, Cu and Mg and an aluminium vacancy (VAI). In the S-W plot all points are located on a line parallel to the Al-Cu line intersecting the Al-V<sub>Al</sub> line near the S,W point associated with the vacancy. Clearly, the observed trends are indicative for the generation of defects associated with (uncommitted) Cu solute atoms. Figure 3 focuses on the results obtained for samples exposed to the lowest stress level ( $\sigma_4$ ). Here we noted that during the first half of the lifetime (<400 kcycles) the S,W points shift into the direction of a Cu rich positron trapping site, both after cycling and the subsequent annealing. Only in case the number of cycles approaches the failure regime the defects that are generated have a more vacancy type character. This trend is also observed at the lower stress level  $\sigma_3$ . For higher stress levels  $\sigma_1$  and  $\sigma_2$  the early failure of the samples did not provide enough data points to draw conclusions.





**Figure 1.** Lifetime results for R=0.1 for the T3-C and **Figure 2.** S-W plot obtained from Doppler T3-A material. The number of cycles after which the DB tests were carried out are marked by asterisks.

Broadening tests. Note: The S,W point for Cu and Mg lie outside the range of axis.

### 4. Discussion

The fatigue loading results are compared with the ones obtained by Hautakangas et al. [1,5] for stepwise static tensile loading of T3 and under aged (UA) alloys obtained by solutionizing T3 samples at 495 °C for 30 minutes in N2 gas followed by quenching in ice water and subsequent artificial ageing at 195 °C for 5 minutes. The static deformation of T3 material only shows an increase of S and decrease of W for strain levels above 2.3 % (see figure 4) corresponding to stress levels above the yield strength. Clearly, open volume defects are created. Ageing at RT after the deformation step shows no further change in S and W indicating that the defects are stable [5]. For the UA material, in which a significant amount of Cu is still in solution, static deformation (to 3% strain) again results in the



Figure 3. Experimental S-W paths obtained for  $\sigma_4$  stress levels.

creation of defects, but in this case ageing at RT leads to formation of additional Cu precipitates.

In the present fatigue experiments on T3 material the maximum stress level applied stays below the yield point and hence only minor changes in S and W parameters are expected. However, for samples tested at low stress levels a trend is visible where the data shifts into the direction of the S,W point for Cu. Since RT ageing can not explain this behavior we conclude that during cyclic loading the defects and dislocations together with the limited amount of Cu present (of the order of 0.1%) have formed additional positrons trapping sites. This process does not involve all the defects generated during the fatigue testing because a subsequent annealing treatment at 65 °C moves the S.W point further into the direction of positron trapping sites associated with Cu. Only in case the loading has resulted in sample failure the data shifts into the high S, low W direction, as the plastic zone surrounding the crack tip must have passed through the sample volume probed.

In order to compare the results of the static (figure 4) and fatigue loading experiments (figure 2) we define  $D = \sqrt{(S - S_o)^2 + (W - W_o)^2}$  as the distance of an S,W point to the S<sub>o</sub>,W<sub>o</sub> point of the as prepared T3 sample. In figure 5 D is plotted as a function of the calculated real stresses. Cyclic loaded samples that were not broken all have a value of D close to zero irrespective of the load level. This is not unexpected since the fatigue stress levels are in the elastic region and damage evolution is less wide spread. As pointed out earlier, in broken samples local plastic deformation in a region around the crack, which is probed in the PA test, has occurred. For theses samples D has the same magnitude as the strained ones. However, as the amount of local deformation can not be quantified uniquely, care should be taken in quantifying the degree of damage accumulation and interpreting differences between samples.



**Figure 4.** S and W parameters as a function of the total strain. Data from [5].



**Figure 5.** Comparison between static and dynamic loads in terms of maximum real stresses and D.

### 5. Conclusion

While the damage accumulation of AA2024 T3 material during plastic straining has been demonstrated convincingly, only weak PA spectrum changes have been recorded for samples loaded dynamically. More detailed PA measurements are therefore required to measure fatigue damage evolution and its disappearance by intermittent low temperature annealing treatments.

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The authors acknowledge the financial support of the IOP Self Healing Materials (the Netherlands) in the form of project SHM08736, "Development of Self Healing in precipitation hardened and dispersoid strengthened aluminium alloys and determination of their fatigue properties".