# Experimental report

22/11/2016

**Proposal:** **1-02-119**

**Council:** 4/2012

**Title:** Cold compression of heat treated aluminium alloys and investigating the associated {311} peak broadening.

**Research area:** Materials

**This proposal is a new proposal**

**Main proposer:** **Christopher TRUMAN**

**Experimental team:** Jeremy ROBINSON

**Local contacts:** Thilo PIRLING

**Samples:** Al

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| SALSA | 5 | 5 | 03/12/2012 | 08/12/2012 |

**Abstract:**

This experiment will aim to measure the size and distribution of residual stresses remaining in heat treated 7449 aluminium alloy rectilinear blocks bodies when cold water quenched and subsequently stress relieved using cold compression. The amount of cold compression will be systematically varied from 0% to 5%. Six rectilinear blocks will be characterised, each with a sacrificial pair used for a stress free sample. The {311} diffraction peak will be utilised for all measurements and the characteristics of the peak assessed to quantify the influence of plastic deformation on peak broadening. The aim of the experiment is twofold. The first objective will be to characterise the efficacy of cold compression on residual stress, the second to determine the nature of the relationship of peak broadening with plastic deformation.

**Cold compression of heat treated aluminium alloys and investigating the associated {311} peak broadening.**

**Experiment number 1-02-119 Date of report 22 November 2016**

Abstract

The residual stresses in heat treated 7075 aluminium alloy blocks have been characterised using SALSA. The influence of uniaxial cold compression on relieving the residual stress has been determined. Systematically increasing the magnitude of cold compression from 1 to 10% has been shown to have a small beneficial effect on the final residual stress distribution, by reducing the magnitude of the range in the triaxial residual stresses. The effect of an overaging precipitation treatment on the residual stress has also been characterised, and this was found to have a significant stress relieving influence (25-40%). A relationship between the width of the 311 diffraction peaks and the amount of cold compression was also observed.

Introduction

The heat treatment of precipitation hardened aluminium alloys has long been known to introduce high magnitude residual stresses. Severe thermal gradients arise when these materials are immersion or spray quenched from the solution treatment temperature (470°C) using cold water. At the solution treatment temperature, aluminium alloys are mechanically very soft and remain so until the temperature has fallen substantially. The hardness (and by inference, yield stress) rapidly drops when the testing temperature rises above 200°C. The thermal gradients and corresponding thermal strains arising during quenching can therefore easily induce inhomogeneous plastic flow to occur. Upon completion of cooling to room temperature, this can result in distortion, including deflection and twisting, cracking and/or residual stresses. The distortion and final stress pattern reflects the geometry of the component and of the temperature gradients generated during the quench. When aggressive quenching is used, for example immersion in cold water, the residual stresses are normally so large (>200MPa) that most aerospace products must be post quench stress relieved using plastic deformation, applied by stretching or cold compression. One aim of this investigation is to quantify the through thickness residual stresses after the application of cold compression in rectilinear aluminium blocks made from 7075.

Cold compression of large rectilinear aluminium alloy forgings is a long established industrial method for reducing residual stresses. Commercial practice involves application of around 2% plastic strain. This amount of plastic strain has only a small impact on the final geometry. The mechanical strength of the material in the direction of the applied plastic deformation can be influenced by the Bauschinger effect (weakening of the material when it is deformed in the reverse direction after a plastic strain in the forward direction). What does remain elusive is good quality data that confirms this as the most appropriate strain magnitude, when applied by compression (or stretching). Application of plastic deformation also leads to peak broadening during diffraction caused by non-uniform strain and a reduced effective crystallite size If it can be demonstrated that there is a systematic relationship between plastic deformation and diffraction peak widths, this could be a useful quantitative characterisation tool. For example, to quantify the amount of plastic deformation that occurs during the imposition of thermal gradients, when quenching in different media, or media at varying temperatures. Experimentally, this information is difficult to elucidate and is currently estimated from numerical modelling techniques. In this investigation. six 7075 blocks cut from 82 mm thick hot rolled plate, were heat treated and immersion cold water quenched prior to cold compression. Cold compressions of 0, 1.4, 2.0, 4.6, 7.6 and 9.9% were applied to samples in the short transverse (ST) direction of the original plate. Samples were then over aged into the fully heat treated condition. Neutron diffraction measurements were made on all samples to determine the residual stress distribution remaining after cold compression and aging. Duplicates samples were manufactured to provide strain free references. Spare blocks were also used to fabricate cylindrical cold compression "calibration" samples and material for microstructural characterisation. The cylinders were plastically strained in the ST direction to a range of plastic strain levels up to 44%. The aim was to attempt to produce an approximate stress-strain curve linked to broadening of the {311} matrix diffraction peak.

Material details and procedures

7075 hot rolled plate was supplied by Mettis Aerospace UK. The plate was 82 mm thick (short transverse-ST direction). Samples of size 120 mm (rolling direction-L, x), 44 mm (transverse direction-LT, y) and 82 mm (ST, z) were cut and machined from the plate. Solution heat treatment was 2 hours at 470°C followed by cold water quenching (TW<20°C) with vigorous agitation. Each block was quenched individually with the ST direction vertical as the block entered the water. Cold compression followed immediately (to minimise the influence of natural aging), and was applied in a single pressing in the z (ST) direction on a 250 tonne hydraulic press at a crosshead speed of 20 mm $s^{-1}$. The amount of compression was controlled using steel stop plates of appropriate thickness. The load bearing surfaces (x-y or L-LT) were lubricated with a light oil. The coefficient of friction during cold compression, μ, determined using ring compression tests was in the range 0.15 to 0.17.13

After cold compression, all the blocks except B8 and B7 were artificially aged for 7 hours at 105°C followed by 9 hours at 175°C. This is a normal over age that renders the material much stronger than the as quenched condition, but also confers good stress corrosion cracking resistance. Eleven solid cylinders for the peak broadening experiment were cut from the plate, with the cylindrical axis corresponding to the ST direction. These were solution treated for 45 minutes at 470°C, cold water quenched and then cold compressed on a 500kN load frame after a post quench delay of less than 15 minutes. Cold compression varied from 0 to 44%. Cylinders were then overaged after cold compression.

Neutron diffraction measurements

SALSA was used with monochromatic radiation of approximate wavelength 1.6Å. The position of the {311} aluminium peak was determined. A sampling gauge volume of 2 x 2 x 2 mm3 as defined by the incident beam slit width and height, and the diffracted beam radial collimators was used. The gauge volume was estimated to contain about 1000 grains. The blocks were positioned on the instrument stage to permit measurements of strains in the three original primary working orthogonal directions. These directions were assumed to be the principal stress directions, being coincident with the direction of maximum heat flow out of the block surfaces during quenching. The measurements originated from the vertex at the centre of the blocks, moving out to the faces with the directions following the primary mechanical working directions. Strain measurements were made at discrete points along each line. Strain free reference prisms (15 (x, L) x 10 (y, LT) x ~82 (z, ST) mm) were extracted from the centre of certain duplicate blocks by electro-discharge machining. Strains were measured along the long central axis (ST, z direction) of these prisms. Lattice spacings were converted to residual strains and stresses using the standard three dimensional Hooke's law. A Young's modulus (E) of 70 GPa and a Poisson's ratio (ν) of 0.3 was used in all the calculations. These elastic constants have been found by the authors to offer the best agreement between neutron diffraction and other residual stress measurement techniques, including x-ray diffraction, incremental centre hole drilling and deep hole drilling for 7000 series alloys. Multiple (repeatability) neutron diffraction measurements on the blocks and the associated stress free samples allowed an estimation of one standard deviation random uncertainties as ± 30 MPa. These uncertainties were much larger than the peak fitting errors. The cylinders were measured at a single point at their centres.

Results

The residual stresses present in Block B9 (cold water quench CWQ + overaged OA) which received no cold compression are shown in Figure 1. Results from two instruments are shown on this figure (SALSA and POLDI at SINQ) with additional x-ray surface residual stress measurements made using the sin2ψ technique. The strain free reference was extracted from block B1. The three figures correspond to line scans in the x (L), y (LT) and z (ST) directions starting at the block centre. The residual stresses measured on both instruments were in good agreement. The distribution of residual stress followed the typical pattern of compressive in the surface and tensile in the core (the component of stress in the y (LT) direction in the centre of the block was close to zero for this geometry). The magnitudes of these stresses were significantly lower than stresses measured in other *unaged* 7000 series alloy samples of similar size. A typical figure of stress relief was 25-40%. This is comparable to that observed in over aged 7050 rectilinear blocks of similar geometry.

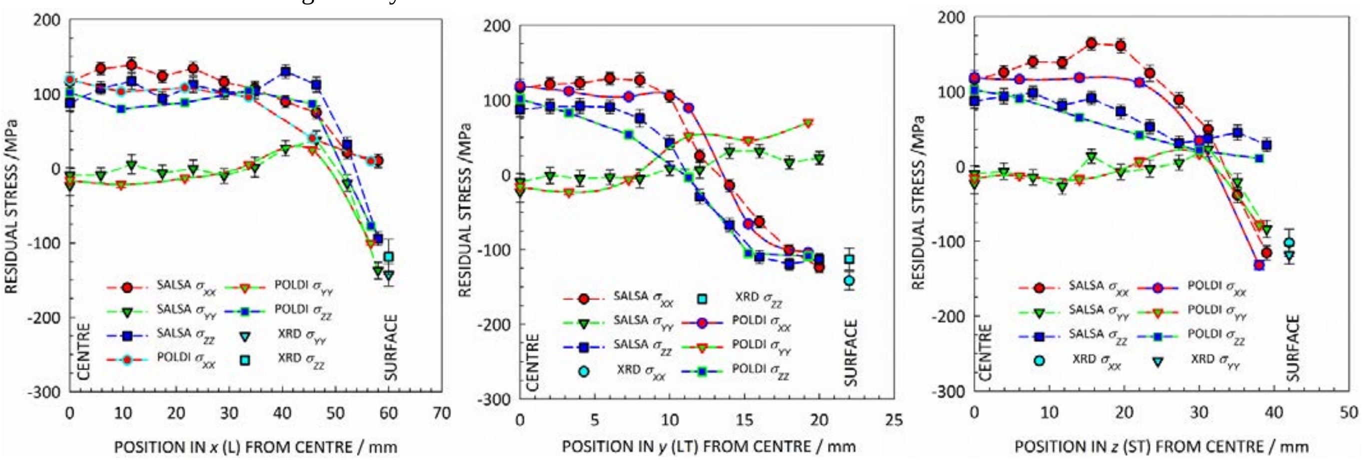



**Figure 4. Residual stresses present in the three orthogonal directions of Block B9. Block had been cold water quenched and overaged. The results from two neutron diffraction instruments are shown in the figure with surface x-ray measurements. Errors bars correspond to peak fitting errors. Repeatability uncertainty ± 30 MPa. (SALSA and POLDI).**

Block B2 was cold compressed by 9.9%. After subsequent over aging, the residual stresses remaining are shown in figure 2. This figure compares residual stress from the x (L) scan to those that would be present without cold compression (B9). It is clear just how effective cold compression is in reducing and homogenising the residual stresses through the thickness of the block.

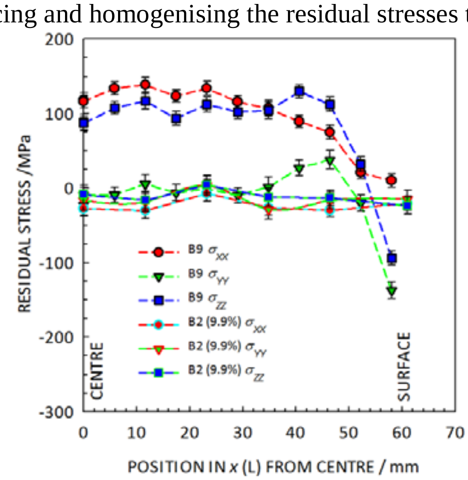



**Figure 2 Comparison of residual stresses remaining in a block cold compressed 9.9% (B2) and a block receiving no cold compression (B9) for a line scan in the longitudinal direction (x) of the blocks. (SALSA).**

Figure 3 displays the residual stresses from the x (L) scan from block B6 (1.4%) and block B2 (9.9%). The illustrates that even when the amount of cold compression is only 1.4%, the cold compression process is still very effective.

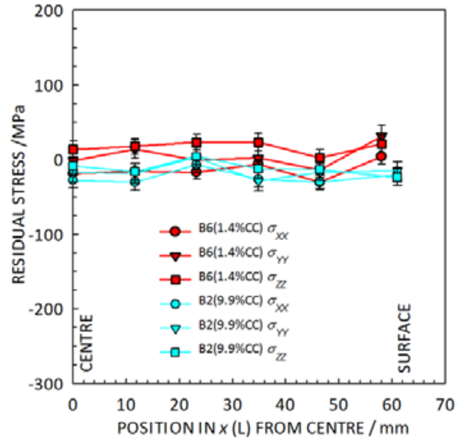
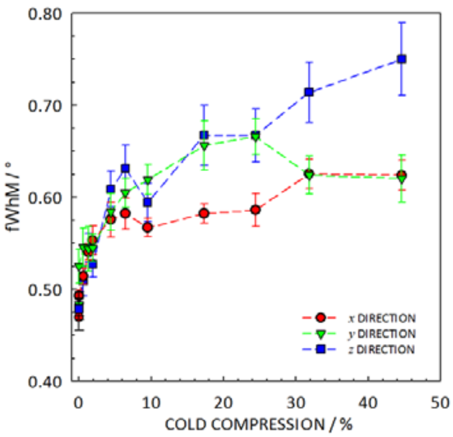
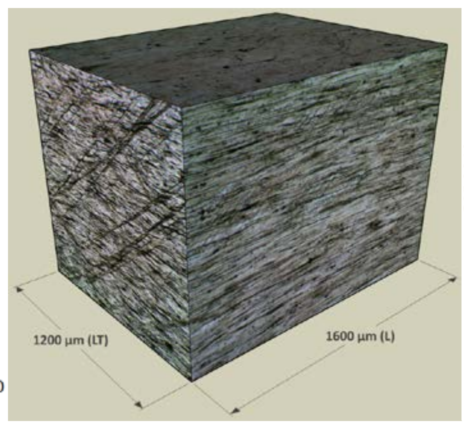



**Figure 3. Comparison of blocks cold compressed by 9.9% (B2) and 1.4% (B6). (SALSA).**

The full width half maximum (fWhM) 311 peak widths from the deformed cylinders are indicated in figure 4. Peak widths were measured for three orthogonal directions of the cylinders with the same coordinate system as that for the blocks. The peak widths increased rapidly for cold compressions up to 6.4%, but then changes were more erratic. In most cases the peak widths continued to increase but it was not a systematic change. A possible reason for this is the increasing trend towards shear band formation during plastic deformation localising the deformation.



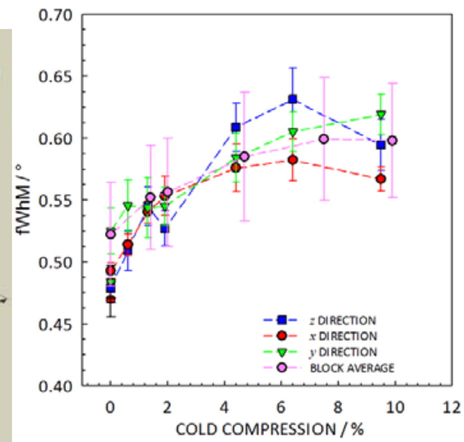




**Figure 4 (left). Full width half maximum 311 peak widths measured from deformed cylinders. Error bars correspond to peak fitting errors. Figure 5 (middle). Microstructure of the 7075 plate in the as quenched, cold compressed 44% and aged condition. The grains contain evidence of multiple slip and the deformation has localised through the formation of shear bands. Figure 6 (right). Low strain portion of figure 4, with the average fWhM calculated from all observations made on the rectilinear blocks B9, B6, B3, B4, B10 and B2 added. Error bars for the blocks correspond to ±1 standard deviations of the fWhM measurements.**

Certain cylinders were examined metallographically, and up to $\varepsilon = 0.17$ there was minimal barrelling of the cylinders and no evidence of shear band formation. Beyond this strain, shear bands could be detected during plastic deformation leading to localisation of the deformation, figure 5. The shear bands initiated at the edges of the cylinder and then propagated diagonally towards the centre with increasing strain. Away from the shear bands, the deformation was more uniform in all cylinders examined. This was manifest as multiple slip within the grains. Slip could not be detected in individual grains at $\varepsilon < 0.17$. When $\varepsilon = 0.44$, the shear bands were well defined and extended to the centre of the cylinder.

The fWhM of 311 peaks measured from the rectilinear blocks B9, B6, B3, B4, B10 and B2 are superimposed on the cylinder data in figure 6. Here the trend can be seen to be the same and the peak widths increased with strain. In this case the fWhM data is a simple average of all measurements made on the blocks. This increases the uncertainty in the data. In addition, it was noted that heavily strained cylinders became oval, indicating the presence and role of crystallographic texture on the deformation mechanism. In summary, the trend observed in the cylinder peak width data does correspond to that seen in the blocks, but once the strain becomes inhomogeneous the correlation cannot be maintained.

## <u>Conclusions</u>

Cold water quenching 7075 from 470°C induces large magnitude residual stresses that vary from tension in the interior to surface compression. Over aging the 7075 typically reduces the residual stresses by 25-40%. Cold compression reduces the residual stresses to very low magnitudes and within the uncertainty of the neutron diffraction measurement technique. Cold compressions of 1.4% alleviate the bulk of the residual stresses but greater cold compressions appear beneficial in reducing the range of the remaining residual stresses. The fWhM 311 peak widths increase with deformation and the measurements in small cylinders are in broad agreement with those measured in the rectilinear blocks.