Cold compression of 7075 and factors influencing stress relief

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Abstract
The residual stresses in heat treated 7075 aluminium alloy blocks have been characterised using neutron diffraction, x-ray diffraction and incremental centre hole drilling. Samples were quenched to induce high magnitude residual stresses which were then stress relieved by the controlled application of plastic deformation using a cold compression technique. The experimental variables investigated were the quench water temperature, and the post quench delay. This delay is considered to influence the final residual stress magnitudes because of hardening precipitation occurring by natural aging during the period between quenching and the application of plastic deformation. Cold compression significantly lowered the residual stresses in all samples. Neutron diffraction measurements demonstrated a benefit to applying plastic deformation as soon as possible after quenching. However, this beneficial effect was not evident when characterising surface residual stresses using hole drilling or x-ray diffraction.

Introduction
The era when aluminium alloys dominated large passenger aircraft structures now appears to be ending. Unless the cost of ownership and repairability issues surround the use of carbon fibre composite become untenable, aluminium alloys are set to become an important but secondary material in the aircraft designers’ handbook. One aspect of aluminium alloy processing that has accelerated the move to composites is the problem of shape distortion during machining operations.\textsuperscript{1,2} These distortions are driven by residual stresses, which arise from the necessity to rapidly cool precipitation hardened alloys as part of the heat treatment step, necessary to develop high strengths.\textsuperscript{3,4}
In this investigation, the residual stresses in heat treated 7075 aluminium alloy blocks have been characterised using two neutron diffraction strain scanning instruments. High magnitude residual stresses arise during quenching from the solution heat treatment temperature. The influence of uniaxial cold compression (or stretching) on relieving these residual stresses is an established technology. One of the factors affecting the efficacy of the cold compression process is the time delay between quenching and cold compressing the material, although this has received almost no attention from the academic community. The different cooling regimes that occur in the surface and core of a large aluminium alloy component mean that metallurgical differences exist and thus different volumes may respond differently to subsequent aging and work hardening processes. This is especially true if the alloy is quench sensitive. In this investigation 7075 blocks, (7075 is an alloy known to be quench sensitive) were subject to post quench delays of either 30 minutes or 240 minutes prior to a single application of approximately 1% cold compression. X-ray, neutron diffraction and incremental centre hole drilling techniques were used to characterise the residual stresses in the blocks before and after cold compression.

**Experimental details**

**Material, heat treatment and cold compression**

Samples of the Al-Zn-Mg-Cu alloy 7075 were supplied by Mettis Aerospace, UK in the form of hot rolled plate. The plate was 82 mm thick (short transverse-ST direction). Rectilinear blocks of size 120 mm (rolling direction-L, x), 44 mm (long transverse direction-LT, y) and 82 mm (ST, z) were cut and machined from the plate. Heat treatment included solutionising for 2 hours at 470°C, followed by water quenching with vigorous manual agitation. The water temperature was nominally 60°C but for certain samples, cold water (T<20°C) was used. Each block was quenched individually with the ST direction vertical as the block entered the water. After quenching, samples were either aged for 7 hours at 105°C or cold compressed after a set period known as the post quench delay and then aged, as shown in Table 1.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Strain scanner</th>
<th>Water temperature °C</th>
<th>Post quench delay Minutes</th>
<th>Cold compression %</th>
<th>Aging treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>A5</td>
<td>POLDI/E3</td>
<td>60</td>
<td>0</td>
<td>0</td>
<td>7h at 105°C</td>
</tr>
<tr>
<td>A9 (Dzero)</td>
<td>POLDI/E3</td>
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<td>0</td>
<td>0</td>
<td>7h at 105°C</td>
</tr>
<tr>
<td>A10</td>
<td>POLDI</td>
<td>60</td>
<td>30</td>
<td>1.2-1.6</td>
<td>7h at 105°C</td>
</tr>
<tr>
<td>A6 (Dzero)</td>
<td>POLDI</td>
<td>60</td>
<td>30</td>
<td>1.5-1.7</td>
<td>7h at 105°C</td>
</tr>
<tr>
<td>A7</td>
<td>POLDI</td>
<td>60</td>
<td>240</td>
<td>1.1-1.6</td>
<td>7h at 105°C</td>
</tr>
<tr>
<td>A1 (Dzero)</td>
<td>POLDI</td>
<td>60</td>
<td>240</td>
<td>1.2-1.6</td>
<td>7h at 105°C</td>
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<td>0</td>
<td>0</td>
<td>Natural age</td>
</tr>
<tr>
<td>B7 (Dzero)</td>
<td>POLDI</td>
<td>20</td>
<td>0</td>
<td>0</td>
<td>Natural age</td>
</tr>
</tbody>
</table>

Table 1. Details of cold compression and aging for 7075 sample blocks

Blocks were cold compressed by a single application of force 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, \( \mu \), determined using ring compression tests was in the range 0.15 to 0.17.\(^{17}\)

The microstructure of the 7075 plate in the as quenched condition consisted of long, pancake type grains. The grains were 50-100 µm thick (z, ST) by 150 - 200 µm wide (y, LT) with a length of 500-1000 µm (x, L). The grains did not contain an optically resolvable substructure and did not appear to have undergone any recrystallisation type events. There was no evidence of second phase precipitation (\( \eta^-\text{MgZn}_2 \)), but the expected normal coarse constituent phases were present. The intragranular precipitates arising after aging (GPZ, \( \eta' \) and \( \eta \)) were not resolvable in the optical microscope.

**Residual stress characterisation.**

**Neutron diffraction**

Measurements were made following the guidelines present in recently published papers.\(^{18-20}\)

Neutron diffraction was performed on two neutron diffraction strain scanning instruments. Residual strains were measured using the time-of-flight neutron diffractometer POLDI (Pulse-OverLap Diffractometer) located at SINQ at the Paul Scherrer Institut in Villigen, Switzerland. POLDI is a time-of-flight instrument, so the positions of multiple diffraction peaks with coincidental scattering vectors were measured. This permitted the use of a full diffraction pattern type analysis.\(^{21}\) The full pattern refinement leads to the determination of an average lattice parameter, based upon the position of all available peaks. The refinement enabled the calculation of the lattice parameter using reflections from up to five sets of interplanar spacings (mainly the, \{200\}, \{400\}, \{311\}, and \{422\} although this was sample and orientation dependent).

Neutron diffraction was also performed on the strain scanning instrument, E3 (HZB, Wannsee, Berlin, Germany). This was used with monochromatic radiation of approximate wavelength 1.47Å. The position of the \{311\} aluminium peak was determined.

A sampling gauge volume of approximately 2 x 2 x 2 mm\(^3\) as defined by the incident beam slit width and height, and the diffracted beam radial collimators was used for both instruments. 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. Figure 1 indicates the dimensions of the block and the dotted line describe the neutron diffraction line scans.
Figure 1. Sample geometry used for residual stress characterisation after water quenching and cold compression. The as-received hot-rolled plate was 82mm thick (short transverse-ST direction, z). Samples of size 120mm (rolling direction-L, x), 44mm (transverse direction-LT, y) and 82mm (ST) were extracted from the plate. Cold compression was applied in the ST direction. The block centre is the origin of the x, y, z coordinate system. Dotted arrows describe the neutron diffraction measurement line scans.

Strain free reference prisms (15 (x, L) x 10 (y, LT) x ~82 (z, ST) mm) were extracted from the centre of duplicate blocks by electro-discharge machining. Strains were measured along the long central axis (ST, z direction) of these prisms. No systematic variation of the strain free lattice parameter was detected in the reference 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.

**X-ray diffraction**

Residual stress measurements using a Sin²ψ technique were performed on a Philips X’Pert x-ray diffractometer using Cu Kα radiation operating in the ω configuration. The measurement procedures followed were those documented in the literature and the best practice guide published by the NPL, UK. The position of the peak arising from diffraction from the aluminium matrix {422} plane was measured (136°<20<139°). Sixteen scans were performed for each stress measurement using equally spaced ψ values within the range 0≤ψ≤60° (positive tilting only, ψ - angle between the surface normal and the bisector of source and diffracted x-ray beam). The resulting spectra were analysed using Philips PC-Stress Software (Version 2.61) with peak locations determined using a Pearson VII fitting technique. In all cases, the sixteen peak positions were used to calculate the straight line d_{422} (interplanar
spacing) versus $\sin^2 \psi$ plots. The calculation of residual stress from the measured peak position was made using the established theory. The elastic constants were taken from literature for the {422} planes. The irradiated area was in the form of a line 2 mm thick and 12 mm long. The penetration depth of the x-rays was assumed to be of the order of 100 $\mu$m calculated using reference data. Calibration of the diffractometer was performed using a sample with a “known” residual stress. This sample was a piece of cold water quenched and aged 7010 alloy that had been characterised on multiple diffractometers located in different institutions over a period of 16 years.

**Incremental centre hole drilling**

Near-surface residual stresses were determined by the ASTM E837 standardised method following the direction of guidelines written by NPL, UK. Measurements were performed on blocks having strengths >500MPa. A Measurement Group Type A, EA-06-125RE-120 strain gauge rosette of nominal size 1/8 inch was selected to maximise the sampling gauge volume which was evaluated at 7:4mm$^3$ for the 2mm measurement depth. The drilled hole diameter to mean gauge diameter ratio was calculated at 0.38 from a maximum permitted 0.4 in each case, the upper-level allowing increased strain sensitivity. The installation was accomplished in compliance with Measurements Group Instruction Bulletins B-129-8, and B-127-14 using M-Bond 200 adhesive. The orbital drilling technique was implemented for each stress measurement using a Tungsten Carbide Inverted Cone 1:8mm dental drill burr. A Measurements Group P-3500 Indicator employing Quarter-Bridge temperature-compensation circuitry was used to record the relaxed strains in conjunction with SB-10 Switch and Balance Unit. The device had a resolution of $\pm 1 \mu\varepsilon$, and an accuracy of $\pm 0.5\%$ of reading $\pm 3 \mu\varepsilon$ which meets with the requirements outlined in the test standard. Residual stresses were determined by the ASTM E837 uniform calculation procedure using the specialised H-Drill computer software program. This calculation variant was considered by the authors to be the most technically appropriate for partnership with diffraction determined results as it provides a representative average value of the pre-existing stresses in the total volume of removed material.

**Mechanical testing**

The progress of artificial aging was monitored using Vickers hardness equipment calibrated with a standard test block to the requirements of ASTM E92–92. Electrical conductivity was measured using a Hocking Autosigma 3000DL conductivity meter. Conductivity measurements were made in units of % IACS (International annealed copper standard) where 1% IACS = 0.58 MS.m$^{-1}$.

**Results and discussion**

**Residual stresses arising from quenching into water at 60°C.**

The residual stresses arising from quenching into water at 60°C (with aging for 7 hours at 105°C) are shown in figure 2. These figures illustrate the neutron diffraction measurements made on the POLDI instrument for block A5 using A9 as the strain free reference. In addition, surface hole drilling measurements were made at the centre of the face of the block defined...
by the L and ST directions. The single value uniform stress arising from the hole drilling measurement is presented on the appropriate figure. The data illustrates the block contains an interior tensile stress which turns compressive as the surface is approached. This is typical of the distribution arising from immersion quenching of regular shaped parts into water.\textsuperscript{33} Quenching into water at 60°C is used commercially as this is reported to introduce more uniform residual stress distribution compared to cold water. However, there is limited reliable scientific evidence to support this observation.\textsuperscript{34, 35}

Figure 2. Variation of residual stress in block A5 with position along a line from the block centre towards the block surface. Measured by neutron diffraction on both the POLDI and E3 instruments. Part A) is a line scan in the x (L) direction, part B) is a line scan in the y(LT) direction and part C) is a line scan in the z(ST) direction. A5 was quenched into water at 60°C. The results from two neutron diffraction instruments are shown in addition to results from hole drilling at the centre of the L-ST face in part B).

Comparison of residual stresses in blocks quenched into cold water and water at 60°C.
The use of the higher water temperatures does affect the magnitudes of the residual stresses and this can be seen in figure 3 which compares the residual stresses in block A5 quenched into water at 60°C, with those in the cold water quenched block B8, both measured on POLDI. (B8 did not receive an artificial age but aging at 105°C has little to no influence on the residual stress.\(^{36}\)) The reduction in residual stress arising from quenching into the heated water is inconsistent and varies with location but can be estimated at approximately 15%. The hole drilling data indicated a reduction of 23% in the residual stress in the x (L) direction and a 6% reduction in the y (ST) direction. Little significance can be attributed to these differences as they fall within the experimental uncertainty of the hole drilling measurement techniques. However, the neutron diffraction observations do suggest that quenching in hot water does produce a small benefit in lowering the residual stress magnitudes. X-ray measurement were made on the surface of block B8 corresponding to locations at the end on the neutron diffraction line scans. The x-ray values presented in Table 2, were in line with the neutron diffraction measurement when these were extrapolated to the sample surface. Multiple repeat x-ray measurements were made at each location and the values are the average of these measurements.

<table>
<thead>
<tr>
<th>Block B8 (CWQ) XRD</th>
<th>Residual stress</th>
<th>Peak fit error</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MPa</td>
<td>MPa</td>
</tr>
<tr>
<td>A)</td>
<td>$\sigma_{xy}$</td>
<td>194</td>
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<tr>
<td></td>
<td>$\sigma_{xy}$</td>
<td>189</td>
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<tr>
<td>B)</td>
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<tr>
<td></td>
<td>$\sigma_{yy}$</td>
<td>185</td>
</tr>
<tr>
<td>C)</td>
<td>$\sigma_{xy}$</td>
<td>197</td>
</tr>
<tr>
<td></td>
<td>$\sigma_{yy}$</td>
<td>179</td>
</tr>
</tbody>
</table>

Table 2. Surface x-ray diffraction measurements corresponding to locations at the end of the neutron diffraction line scans which are the surfaces highlighted in Figure 3, parts A), B) and C) for block B8.
Figure 3. Residual stresses in blocks A5 (quenched into water at 60°C) and block B8 (quenched into cold water). Results from hole drilling measurements at the centre of the L-ST face are also shown in part B).

**Influence of cold compression on as quenched residual stresses**

Block A10 was quenched into water at 60°C and then cold compressed after a delay of 30 minutes. The amount of cold compression determined by measuring the block height after removal of the load, was between 1.2% in the centre of the block, to 1.3% when measured at the long edge of the face defined by the x (L) and y (LT) directions, and 1.6% on the short edge. 7000 series alloys naturally age at room temperature, but after 30 minutes the changes in hardness and electrical conductivity (a common industrial measurement technique to monitor the aging process) are insignificant, as shown in figure 4.
Figure 4. Natural aging response of 7075 after quenching into water at 60°C. Vickers hardness (using a mass of 20kg) and electrical conductivity measurements. Error bars are ±3 standard deviations.

The resulting residual stress distribution remaining in A10 after cold compression is shown in figure 5. The ordinate scale in A), B) and C) is the same as figure 2 and 3 to facilitate comparison of the data. The application of uniaxial plastic deformation is effective at lowering all three orthogonal components of residual stress into the range ±30MPa. These observations are consistent with other investigations.\textsuperscript{37-41} The results of the hole drilling measurement are also included on the figure and these observations were consistent with the neutron diffraction observations. X-ray diffraction results from the surfaces of block A10 were not in such close agreement with the neutron diffraction or hole drilling observations and these are included in part D). The main reason for this was poor (422) peak quality caused by broadening due to plastic deformation and low diffraction peak intensities. However, the stresses were in the range normally associated with a close to zero stress state, as confirmed by measuring the residual stress remaining in annealed samples.

Typically, industry uses 2-2½% cold compression when stress relieving aluminium alloy forgings and plate. The data presented here, in line with other studies, suggests it is not necessary to use this much plastic deformation, and lower magnitude cold compressions are also just as effective.\textsuperscript{23, 42} However, the origin of the number 2-2½% is from the 1950s, and its longevity must reflect its worth.
Figure 5. Residual stresses in block A10 (quenched into water at 60°C) and then cold compressed after a delay of 30 minutes. Results from hole drilling measurements at the centre of the L-ST face are also shown, part (B). Part D is part A) replotted with an expanded ordinate axis and includes x-ray diffraction measurements. Errors bars arise from the peak fitting.

Block A7 was treated in the same way as A10 but was allowed to naturally age for 4 hours before the application of cold compression. During these 4 hours, the electrical conductivity decreased by 4%, and the indentation hardness increased by 8%, both due to due to solute clustering and precipitation of transition phases like GP zones. Despite being harder and therefore stronger, it was still possible to cold compress the block to almost exactly the same cold compression (1.1% in the centre of the block, to 1.1% when measured at the long edge of the face defined by the x (L) and y (LT) directions, and 1.6% on the short edge). The resulting residual stress distribution remaining in A7 after cold compression is shown in figure 6. The ordinate scale is the same as figures 1, 2 and 4 to facilitate comparison of the data. From this figure, it can be seen that for the majority of the measurement points the
range of residual stress has increased ($\sigma_i^{\text{max}} - \sigma_j^{\text{min}}$, where $\sigma_i$ and $\sigma_j$ can be the $\sigma_{xx}$, $\sigma_{yy}$ or $\sigma_{zz}$ component of residual stress). This is made more obvious when the data in figures 4 A) and 5 A) are replotted with the ordinate range fitted to the data as shown in part D) of both figures. These data suggest that allowing the material to naturally age prior to the application of cold compression is detrimental to the efficacy of the cold compression process, even when the amount of cold compression applied is the same. The effect is small but it does reproduce observations made in a previous investigation using the aluminium alloy 7050.\textsuperscript{44}

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**Figure 6.** Residual stresses in block A7 (quenched into water at 60°C) and then cold compressed after a delay of 240 minutes. Results from hole drilling measurements at the centre of the L-ST face are also shown, part (B). Part D) is part A) replotted with an expanded ordinate axis and includes x-ray diffraction measurements. Errors bars arise from the peak fitting.
The post quench delay is an important processing parameter when stress relieving aluminium alloy products. The delay is currently controlled, but only to ensure the materials do not harden significantly making the application of cold compression or stretching much more difficult or impossible. It is not known by the authors if the additional benefits of a short post quench delay are appreciated by the aerospace aluminium alloy product manufacturers. To undertake to stress relieve immediately after quenching in an industrial setting is a significant logistical undertaking. The evidence presented here is not overwhelmingly conclusive but there have now been two investigations, on 7050 and now 7075 that demonstrate the benefit. The mechanism involved that promotes more effective stress relief immediately after quenching is complex and must involve the post yield work hardening behaviour of the material. Immediately after quenching and before natural aging progresses, the response of the material is more homogenous in terms of yield and work hardening. As the material ages, through thickness differences arising from quench sensitivity effects will give rise to differing rates of work hardening in the surface compared to the core of the material. It is thought that it is these differences that can lead to increased residual stresses after cold compression. To fully identify the positive attributes of the effect, either a more sensitive residual stress measurement technique must be brought to bear, or experiments conducted that accentuate the differences caused by the post quench delay. For the latter, four-point bending of as-quenched beams is one possibility that will be investigated by the authors in the future. Of more interest to aluminium alloy product manufacturers will be proof the immediate application of cold compression reduces distortion during machining. Further experimentation in the area of mechanical dissection techniques should go some way to proving if it is worth enduring the recommendation of stress relieving immediately after quenching.

Conclusions

1) The residual stress observations made by two independent neutron diffraction strain scanners instruments (POLDI and E3) on a rectilinear 7075 block quenched into water at 60°C are in complete agreement when bearing in mind an experimental uncertainty of ±30MPa.

2) The through thickness residual stress magnitudes on samples quenched into water at 60°C vary from highly tensile in the core regions to highly compressive in the surface.

3) Quenching into water at 60°C reduces the through thickness residual stress magnitudes by approximately 15% compared to quenching into cold water (<20°C).

4) The application of 1.1-1.6% uniaxial cold compression within 30 minutes of quenching reduces the triaxial residual stresses to very low levels ±30MPa

5) A post quench delay of 240 minutes permits hardening by natural aging. The alloy is 8% stronger after this time. If the material is then cold compressed by 1.1-1.6%, the plastic deformation is not as effective in reducing the residual stresses. However, the residual stresses are still reduced to very low levels.

6) The magnitude of the post quench delay effect is small and close to the experimental uncertainty of the neutron diffraction measurement technique, but from the number of measurements made, the results do appear to confirm the effect is real. However, the effect of the post quench delay is not detectable using x-ray diffraction or incremental centre hole drilling.
7) To minimise residual stresses after the application of plastic deformation, it is proposed that the stress relieving operation be carried out as soon as possible after quenching.

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Disclosure statement
No potential conflict of interest was reported by the authors.

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