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Uphill quenching to reduce residual stress in a heat treatable aluminium alloy

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Keywords

Aluminium alloy 7449; heat treatment; residual stress; uphill quenching; neutron diffraction

Abstract

Three rectilinear blocks of the aluminium alloy 7449 were characterised using neutron and X-ray diffraction. One block was heat treated normally and two blocks were subject to uphill quenching from -196°C to 100°C. Boiling water and steam were used to rapidly increase the temperature of the blocks to reverse the thermal gradients introduced by cold water quenching. It was possible to detect the beneficial influence of uphill quenching on residual stress using either fluid. The influence of steam was very effective but localised and limited to the surface in close proximity to the steam jet. For more uniform stress relief, multiple steam jets will be required to ensure the entire surface receives a significant thermal input.

Introduction

Quenching heat treatable aluminium alloys has the unfortunate consequence of introducing large magnitude residual stresses. In thick aerospace product forms (t > 15 mm), residual stress magnitudes of ± 200 MPa are common after cold water quenching from the solution heat treatment temperature.[1] In this condition, subsequent machining can result in distortion or cracking. For uniform rectilinear shapes, application of plastic deformation is used to reduce the residual stresses.[2-5] However, for complex shapes such as die forgings this is much more difficult.[6] For complex critical parts where residual stresses cannot be tolerated, current practice is to reduce the thermal gradients during quenching, but this compromises the mechanical properties. A long established but rarely used technology is uphill quenching (UHQ) which attempts to reverse the thermal gradients encountered during quenching.[7-9] The interest in uphill quenching arises because it has the potential to be applied to complex geometries that cannot be stress relieved economically by mechanical methods. Complex die forgings (and castings) are good candidates. Only very limited data exists that fully quantifies the stress reduction process and how the distribution of residual stresses is changed through the thickness of components. Uphill quenching, despite being labelled a "cryogenic or cold stabilisation" process and therefore treated with suspicion, does reduce residual stresses when applied to as quenched products. It should not be confused with the simple exposure of parts to sub-zero temperatures which has no effect. It is the technological difficulties of applying the sudden increase in temperature in a controlled manner that have limited its more widespread application. There is also the requirement to minimise the amount of natural aging that can take place between the water quench and immersion in the cryogenic fluid. If the material strengthens at this stage, uphill quenching becomes less effective. In this experiment blocks of the alloy 7449 were processed using conventional quenching and uphill quenching techniques. The through thickness residual stress distribution has been characterised using neutron and X-ray diffraction. Both uphill quenching in boiling water and steam result in stress relief. The effect of steam impingement is shown to be very effective in lowering surface residual stress but the effect is very localised and the depth of penetration is shallow.

Experimental details

Materials and heat treatment

Six rectilinear blocks of 7449 were extracted from a large spar like forging, originally triaxially forged from a large round ingot. Individual blocks were 56mm (Longitudinal - L) x 75mm (Long Transverse - LT) x 125mm (Short Transverse - ST) in size and each had a mass of 1.4 kg. Shrouded type K thermocouples of 1.5 mm diameter were inserted into a spare 7449 test block to allow time temperature profiles to be determined during cold water and uphill quenching. The size of the specimen blocks and the thermocouple tip locations are shown in figure 1. T0 and T2 thermocouple tips were located within 2.25 mm of the block surface, while T1 was located at the block centre.

The microstructure of the blocks, consisted of approximately rod shaped grains elongated into the longitudinal direction with a typical grain length being <1000 μm. In the transverse directions, the grain characteristic dimension was <200 μm. Within these grains a substructure was observed consisting of well-defined polygonised equiaxed subgrains. The diameter of the subgrains was <20 μm. Other coarse phases noted were fragmented Al_7Cu_2Fe constituent particles, and a very small volume fraction of undissolved $MgZn_2$. The material can be classified as completely unrecrystallised. The 0.2% tensile proof stress of cold water quenched 7449 measured in small samples is in the range 140 – 150 MPa.

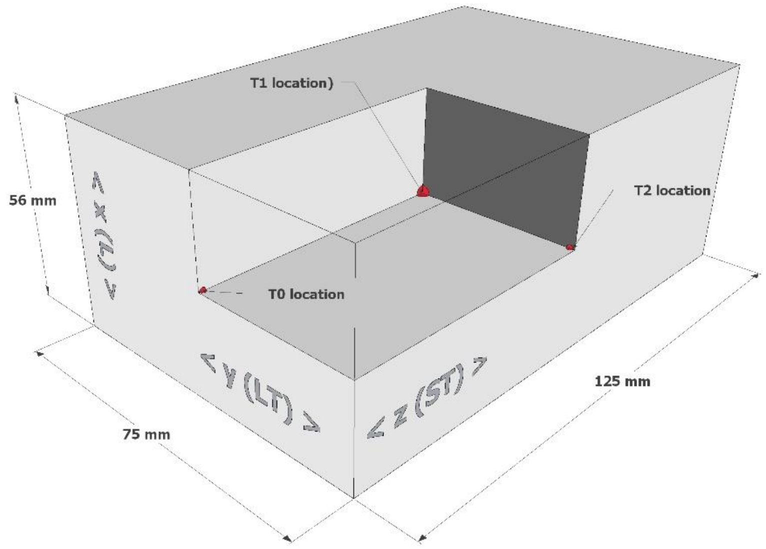


Figure 1. Specimen size and thermocouple locations. Residual stress measurements by neutron diffraction were made on the dark grey face x-y (L-LT).

Heat treatment and uphill quenching

The normal precipitation hardening heat treatment for 7449 includes solution treating at 472°C, followed by cold water (<20°C) immersion quenching and then aging for 6h at 120°C + 10h at 160°C. This procedure was followed but included the UHQ step conducted as soon as possible after cold water quenching. The blocks were cooled to -196°C in liquid nitrogen. As soon as the blocks equilibrated, they were either immersed in a large volume of boiling water or enclosed in a box with two nozzles connected to a 40 kW steam generator capable of delivering dry, or slightly superheated steam, at a nominal flow rate of 0.02 kg s^{-1}. Steam conditions could be varied from atmospheric (100°C at 1 bar) to a maximum of 170°C at 7 bar, depending on the circumstances. In this case, high velocity steam jets at slightly elevated atmospheric conditions were directed onto either side of the x-z (L-ST) faces of the block. A complete thermal profile up to the end of the UHQ is shown in figure 2.

In summary, three conditions were examined, cold water quenched (CWQ) and aged, CWQ+UHQ (boiling water)+aged and CWQ+UHQ (steam)+aged. The over-aging treatment lowers the as quenched residual stress magnitudes, but in 7449 with a maximum aging temperature of 160°C, the influence is small.[10]

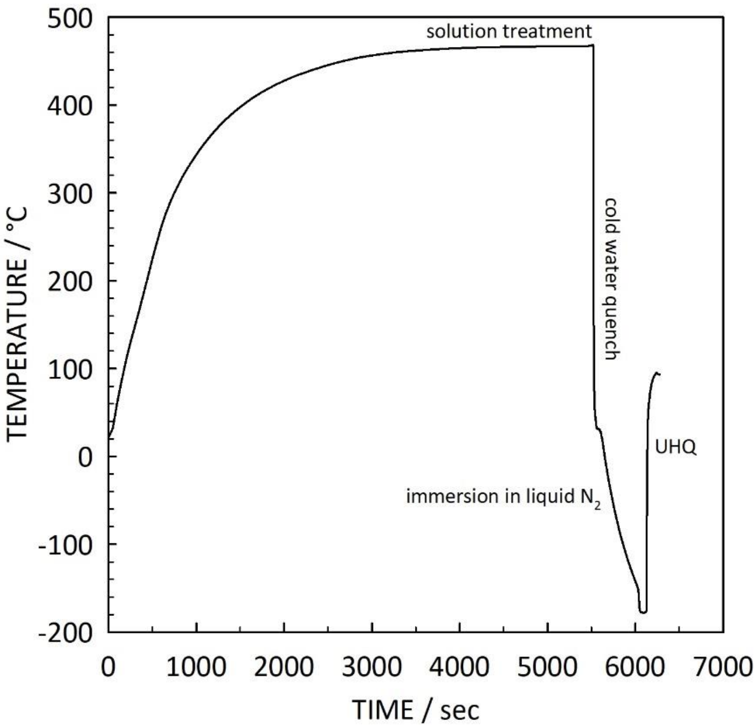


Figure 2. Typical temperature time data including solution heat treatment, cold water quenching, immersion in liquid nitrogen and uphill quenching to 100°C.

Residual stress characterisation

Neutron diffraction

Measurements were made following the guidelines present in recently published papers.[11-13] Neutron diffraction was performed on the strain scanning instrument, STRESS-SPEC (FRM II, Munich, Germany). A take off angle of the Si (400) monochromator was set to produce a beam with a wavelength of approximately 1.67 Å, which defines the diffraction angle of the Al [311] at $2\theta \approx 86.50°$

The sampling gauge volume was approximately 2 x 2 x 2 mm^3 as defined by the incident beam slit width and height, and the diffracted beam radial collimator. The blocks were positioned on the instrument stage to permit measurements of strains on a central quarter plane defined by the x and y directions (L and LT) as shown in figure 1. Strains were measured in all three working directions and these were assumed to be the principal stress directions, being coincident with the direction of maximum heat flow out of the block surfaces during quenching. Strain measurements were made at 40 discrete points on the plane for the CWQ and UHQ (boiling water) blocks, and at 60 points for the UHQ (steam) block.

Each block had a complimentary duplicate subject to the same processing. A strain free reference prism was extracted from each duplicate using wire cutting. This prism had dimensions 8 (x, L) x 75 (z, ST) x 10 mm (y, LT). Multiple orthogonal strain measurements were made long the length of the prism. Lattice spacings were converted to residual strains and stresses using the standard three dimensional Hooke's law.[14] 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.[15, 16] 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 larger than the peak fitting errors. The microstructural induced variation along the length of the strain free reference was small, and encapsulated by the random uncertainties.

X-ray diffraction

Surface residual stress measurements using a $\mathrm{Sin}^2\psi$ technique were performed on a Panalytical X'Pert X-ray diffractometer using Cu Kα radiation operating in the ω configuration. The measurement procedures followed best practice guidelines.[17] The position of the aluminium [422] peak was measured (136°<2θ<139°). Sixteen scans were performed for each stress measurement using equally spaced ψ values within the range $0 \leq \psi \leq 60°$ (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 Panalytical residual stress software (Version 2.3) 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 $\mathrm{Sin}^2\psi$ plots. The calculation of residual stress from the measured peak position was made using the established theory.[18] The elastic constants were taken from literature for the [422] planes.[19] 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 μm calculated using reference data.[18] Calibration of the diffractometer was performed using a specimen with a "known" residual stress. This specimen was a piece of cold water quenched and aged 7010 alloy that had been characterised on multiple neutron and X-ray diffractometers located in different institutions over a period of 19 years. The measurement locations were on the surface of the block on the perimeter of the quarter plane shown in figure 1.

Mechanical testing

The progress of precipitation hardening was monitored using Vickers hardness equipment calibrated with a standard test block to the requirements of ASTM E92–92.[20]

Results and discussion

Cold water quenched only

Cooling from the solution heat treatment temperature by immersion in cold water is rapid, and it is the thermal gradients from surface to core that cause inhomogeneous plastic flow which in turn give rise to residual stress. The cooling curves from two of the embedded thermocouples are shown in figure 3. The surface thermocouple (T1) was located adjacent to a small surface defined by the x and y directions (L and LT); the cooling curve from the large surface was almost identical. The maximum temperature difference between the surface and core was 165°C and this occurred 3.7 seconds after immersion when the surface was at 185°C and the core at 350°C.

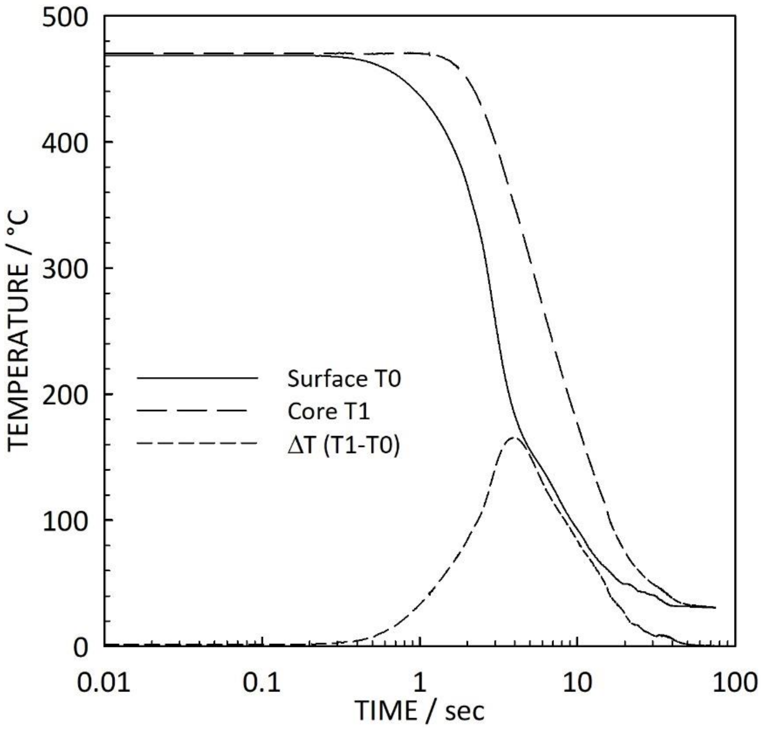


Figure 3. Cooling curves from a cold water quenched block quenched into water at T<20°C. Cooling curves from a small surface (T1) and the core (T0) are shown and the temperature difference between these curves is also indicated (ΔT). Thermocouple locations in figure 1.

The residual stresses within the block are displayed as contour maps in figure 4. The core of the block was in a state of triaxial tension with large tensile stresses occurring in the z (ST) and y (LT) directions. In the x (L) direction the residual stresses were much lower in the interior but turned highly compressive as the x-z face was approached. The distribution and magnitudes of residual stress in the cold water quenched and aged block were typical for this alloy, geometry and treatment. The maximum tensile residual stress measured in the core was a stress in the z (ST) direction and had magnitude 237 MPa. The maximum compressive residual stress close to the surface was a stress in the y (LT) direction and had magnitude -208 MPa. In addition to the neutron diffraction measurement, surface X-ray diffraction measurements were made for certain locations and stress components. These were consistent with extrapolation of the neutron diffraction stresses to the surface.

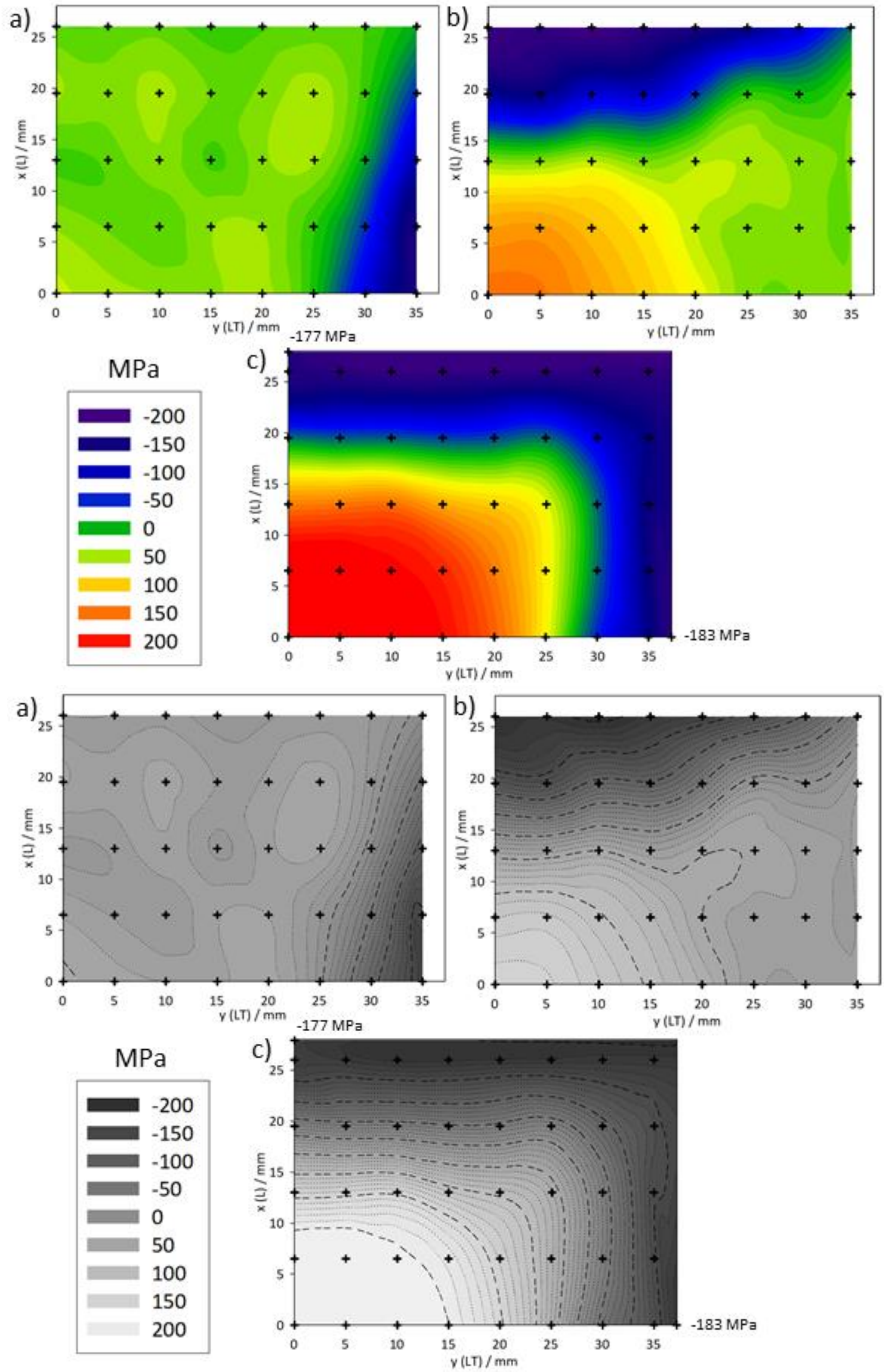


Figure 4. Residual stresses in the cold water quenched block. Part a) Residual stress σ_{xx} (in the x (L) direction). Part b) Residual stress σ_{yy} in the y (LT) direction. Pact c) Residual stress σ_{zz} in

the z (ST) direction. Black crosses indicate neutron diffraction measurement locations. Crosses in the block surfaces are X-ray diffraction measurements. (COLOUR FIGURES ONLINE ONLY)

Cold water quenched and uphill quenched in boiling water

Uphill quenching into boiling water caused a layer of ice to form on the block upon immersion. This slowed the uphill quench. The process was repeated, but the block was manually agitated through the boiling water to accelerate the melting of the ice and increase the rate of heat transfer into the block by forced convection. This halved the duration of the uphill quench with the block interior attaining 0°C after 17 seconds. The heating curves from three embedded thermocouples are shown in figure 5. . Both surfaces heated up at similar rates and faster than the core. The maximum ΔT from small surface to core (T0-T1) was 77°C, when this surface was at 8°C. This occurred 11 seconds after immersion in the water. For the larger surface, the maximum ΔT was 56°C after 9 seconds.

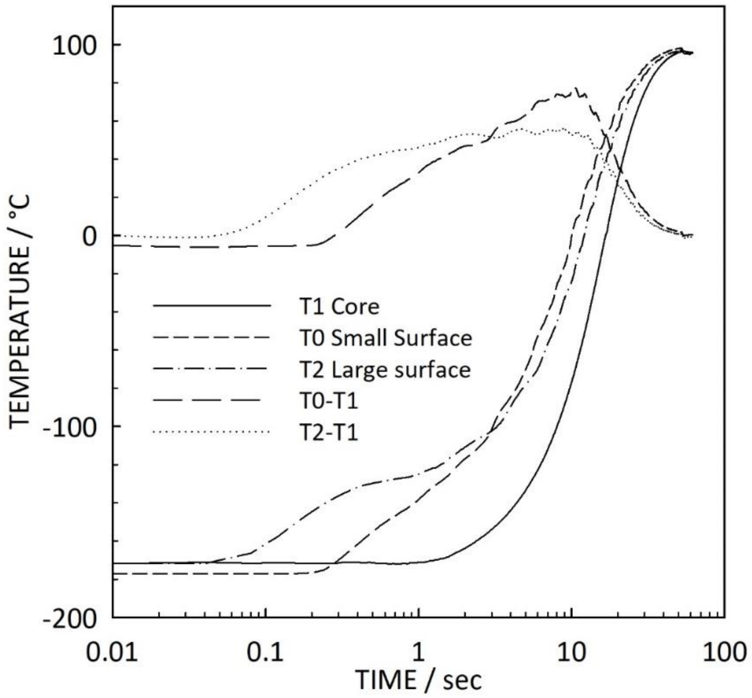


Figure 5. Heating curves from the block during an uphill quench into boiling water with manual agitation. Thermocouple locations in figure 1.

The residual stresses within the uphill quenched block are displayed as contour maps in figure 6. The distribution and magnitudes of the residual stresses were similar to the cold water quenched block. The impact of the uphill quench could be detected and while limited, exceeded the experimental uncertainty. The maximum tensile residual stress measured in the core was a stress in the z (ST) direction and had magnitude 182 MPa. The maximum compressive residual stress close to the surface was a stress in the y (LT) direction of magnitude -137 MPa. Examining the stress change in all measured locations the average stress change was a 20% reduction compared to the CWQ block

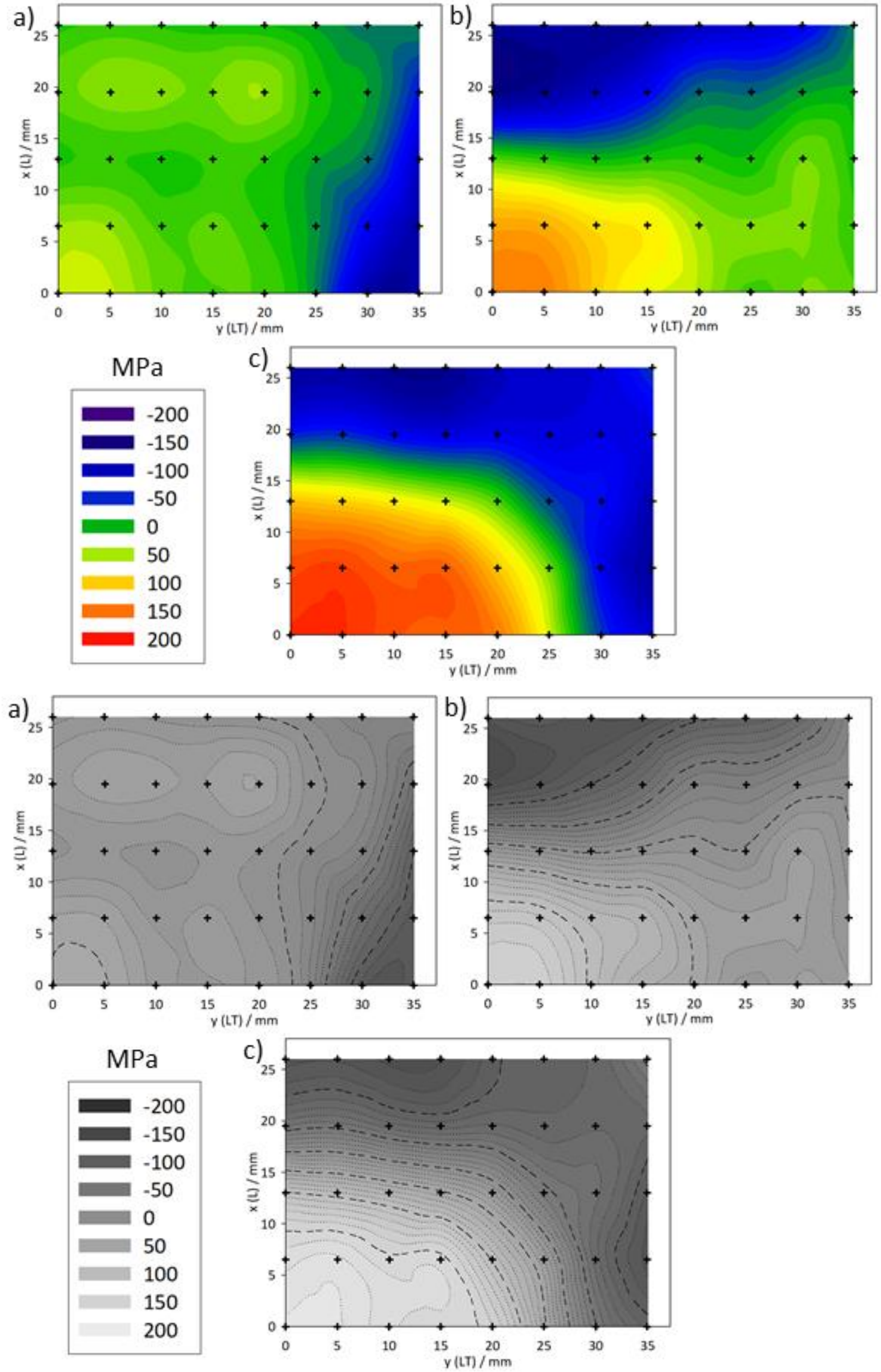


Figure 6. Residual stresses in the cold water quenched block, subsequently uphill quenched into boiling water. Part a) Residual stress σ_{xx} (in the x (L) direction). Part b) Residual stress σ_{yy}

in the y (LT) direction. Pact c) Residual stress σ_{zz} in the z (ST) direction. Black crosses indicate neutron diffraction measurement locations. (COLOUR FIGURES ONLINE ONLY)

Cold water quenched and uphill quenched in steam

Uphill quenching into steam initially caused the steam to condense on the block. Very little water vapour emerged from the box for about 30 seconds. This then increased and water vapour was seen emerging from the box in large volumes. The block interior attained 0°C after 22 seconds. The heating curves from three embedded thermocouples are shown in figure 7. The large face receiving the steam jet heated up quickly and reached 0°C after 6.5 seconds. In contrast, the small face not in direct line with a steam jet heated up at the same rate as the block interior, and only achieved 0°C after 22 seconds. However, the maximum temperature difference between the large face receiving the steam and the core was 164°C after 9.5 seconds

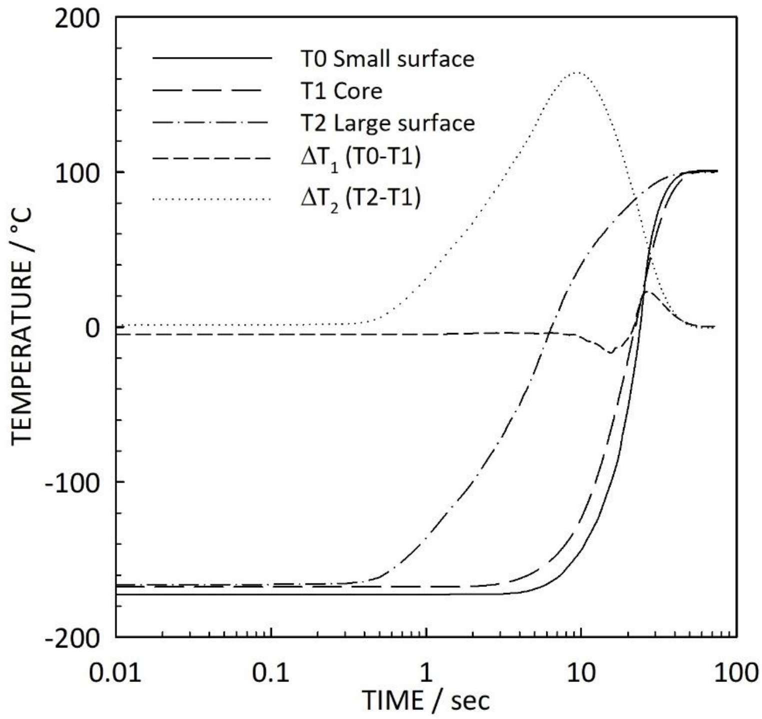
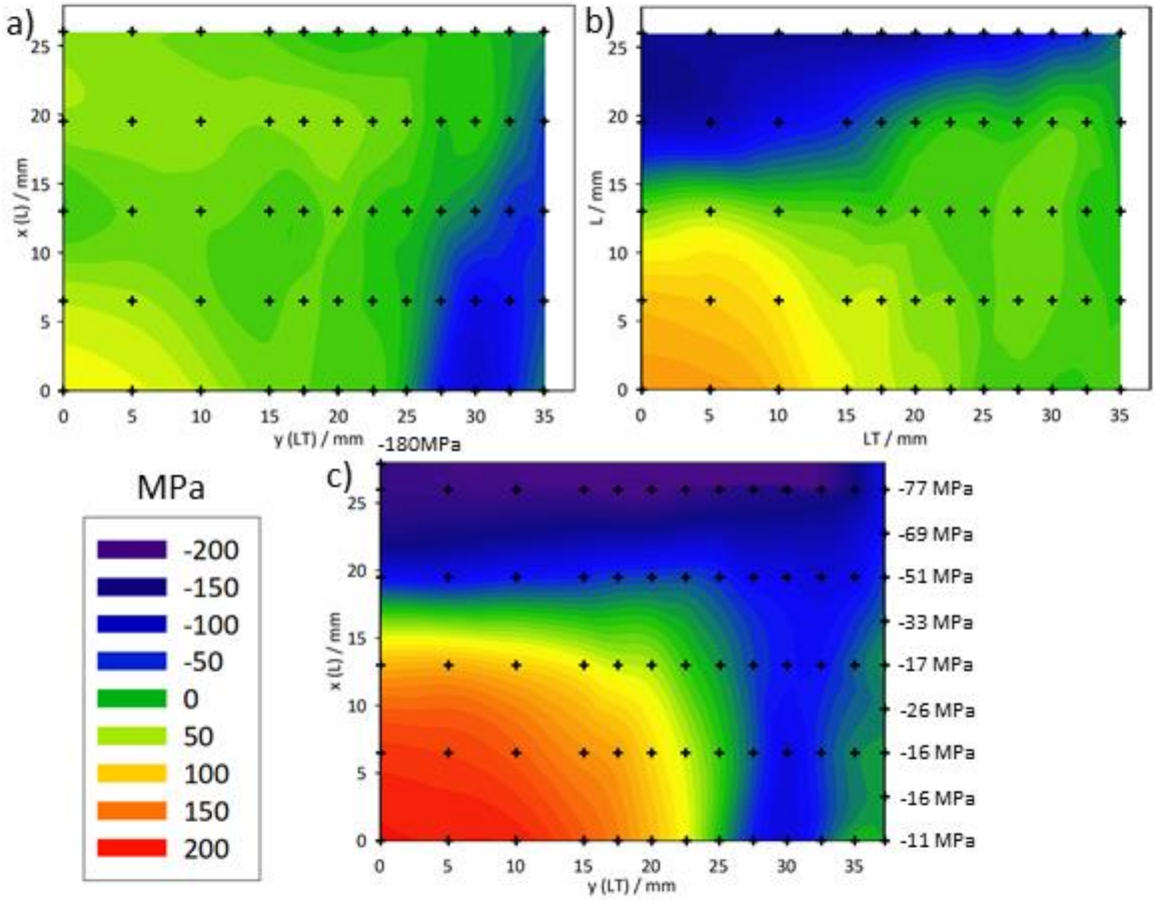


Figure 7. Heating curves from the block during an uphill quench using two steam nozzles. One steam nozzle was adjacent to thermocouple T2. Thermocouple locations in figure 1.

The residual stresses within the block uphill quenched using steam are shown as contour maps in figure 8. X-ray diffraction measurements of the σ_{zz} component of stress at the uphill quenched surface are incorporated into contour map 8 c). Due to limitations of the X-ray diffractometer stage, it was not possible to measure the σ_{xx} component of stress. The distribution and magnitudes of the residual stresses were similar to the cold water quenched block in most locations except where the steam jet had directly impinged onto the side of the block. The maximum tensile residual stress measured in the core was a stress in the z (ST) direction and had magnitude 203 MPa. The maximum compressive residual stress close to the surface was a stress in the z (ST) direction of magnitude -206 MPa. Examining the stress change in all measured locations, the average stress change was a 14% reduction compared to the CWQ block. The impact of the steam can be seen in 8 c). The effect is localised despite the block being completely surrounded with steam within the box. It is clear that the steam has to impinge on the surface to gain significant benefit. However where the steam did impact the sample, the residual stress was almost completely relieved. The depth of stress relief was of the order of 5 mm.



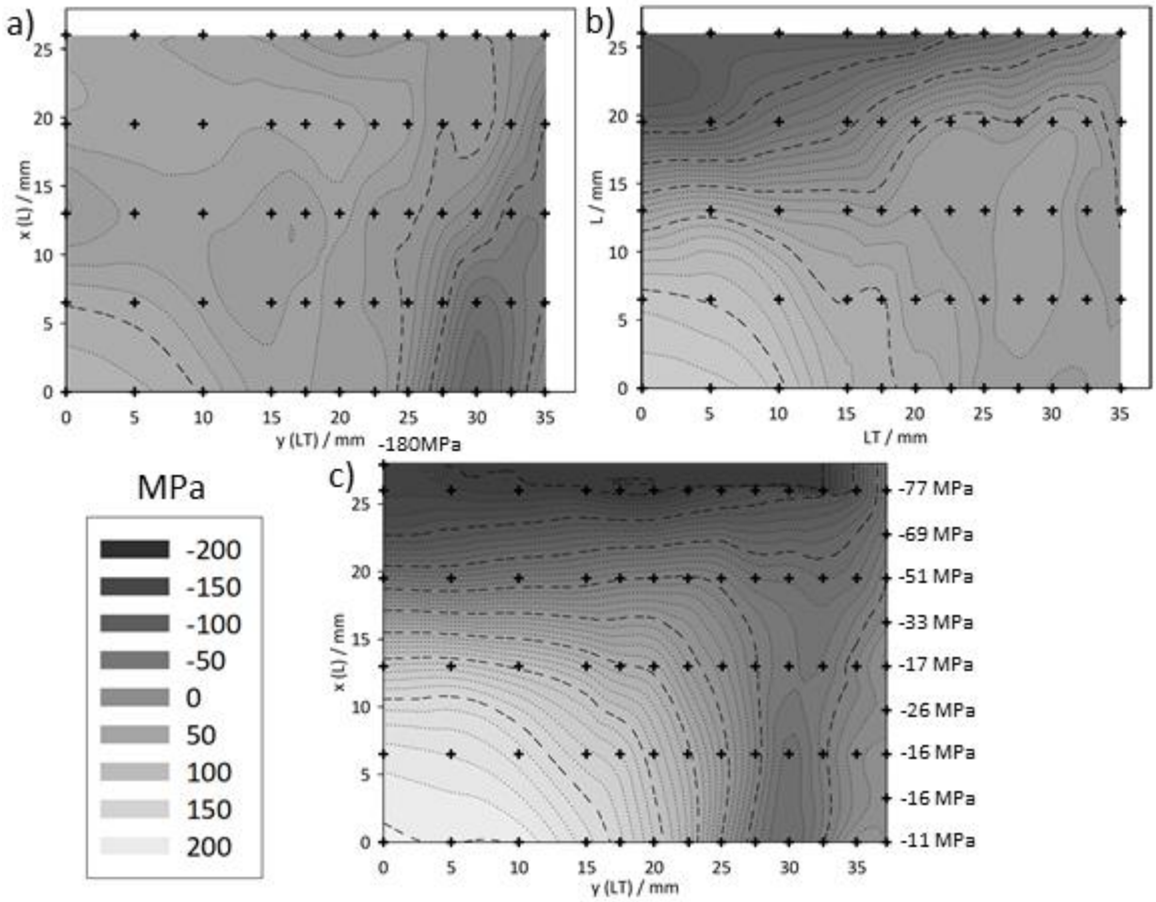


Figure 8. Residual stresses in the cold water quenched block, subsequently uphill quenched using steam. The impingement of the steam jet was at the surface located at y (LT) = 37.5 mm and x (L) = 0 mm. Part a) Residual stress σ_{xx} (in the x (L) direction). Part b) Residual stress σ_{yy} in the y (LT) direction. Pact c) Residual stress σ_{zz} in the z (ST) direction. Black crosses indicate neutron diffraction measurement location, except at surfaces where they are by X-ray diffraction. (COLOUR FIGURES ONLINE ONLY)

These data confirm uphill quenching has the potential to effect significant localised stress relief. This information is not new and the process has been available commercially in the US for many years.[21, 22] Nevertheless, these diffraction results now show how localised the influence of the steam is and the shallow depth of penetration. For this reason, it is clear the process will only be applicable to specific types of component geometries with relatively thin cross sections amenable to the access of jets of steam. What is also clear is the requirement to make sure all the surfaces requiring stress relief be exposed to a jet of steam. The early pioneers of this process were aware of these limitations, but the recurring interest in the uphill quench process arises from the relative low cost and simplicity of the process.[23-27]

Influence of the UHQ process on mechanical properties

Uphill quenching relies on the introduction of non-uniform plastic strains during the temperature increase. The amount of natural aging that can take place between the water quench and immersion in the cryogenic fluid must therefore be minimised. Another constraint is the temperature dependence of the inherent strength of the aluminium matrix as shown in figure 9. Here the solution treated and cold water quenched 7449 alloy was cooled to -196°C and hardness tested as the temperature increased. The heating rate was 12°C/min up to 0°C and approximately 20°C/min above 0°C. This demonstrates that the aluminium alloy becomes stronger at sub-zero temperatures, which does not aid the uphill quench process. It also shows how soft aluminium alloys are at high temperatures, which contributes to the introduction of residual stresses during quenching from the solution heat treatment temperature.

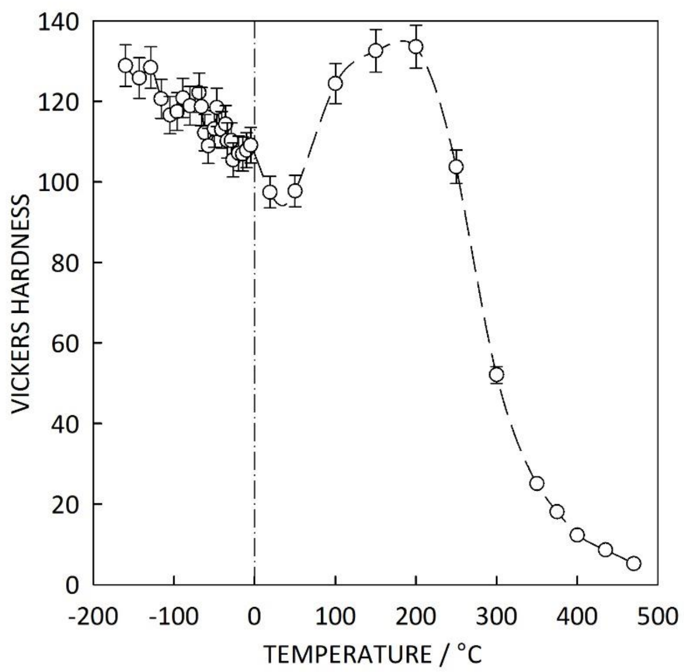


Figure 9 Solution treated and cold water quenched 7449 alloy after cooling to -196°C and being heated back to the solution treatment temperature. The increase in hardness between 100°C and 250°C is due to precipitation hardening.

Another limitation of the UHQ process is the requirement to uphill quench into a fluid at elevated temperature, be it boiling water, steam, or the saturated vapour of a perfluorocarbon compound [28]. Most highly alloyed aerospace alloys age rapidly at elevated temperatures so this could influence subsequent aging treatments. This is shown in figure 10 where it will be noted the hardness increases significantly even after just one minute at 100°C.

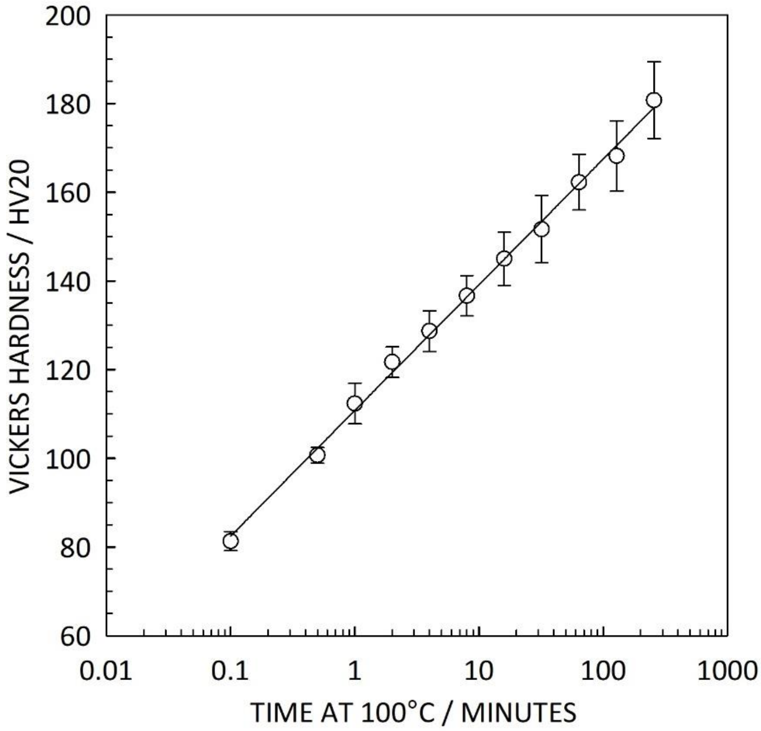


Figure 10 Aging response of 7449 at 100°C

For this experiment, the small specimens were hardness tested immediately after cold water quenching and uphill processing. In the as cold water quenched condition, the blocks had a hardness of 115 HV20. After uphill quenching into boiling water, the hardness increased to 135 HV20. After uphill quenching by steam, the hardness was 130 HV20. However, after subsequent artificial aging (6h at 120°C + 10h at 160°C) all three blocks were 195±5 HV20. This confirms the limited impact of uphill processing on the resulting strength of the alloy 7449.

Conclusions

1. Uphill quenching in boiling water and dry steam have both demonstrated the potential to lower residual stress introduced during the quenching stage of the heat treatable aluminium alloy 7449.
2. Uphill quenching into boiling water from -196°C results in limited detectable stress relief. The change is ~20% but affects most locations uniformly. Uphill quenching into steam at 100°C caused rapid heating of the material adjacent to the steam jet. At these locations, the residual stress was completely relaxed. The effect was localised and other surfaces only demonstrated very limited stress relief which did not exceed 14%. The depth of penetration of the stress relief under the steam jet was 5 mm.

3. A constraint in the process is caused by the alloy being significantly harder when cooled to sub-zero temperatures. 7449 is 60% harder at -196°C compared to the as quenched condition.
4. With multiple steam jets and informed component selection, there is no reason why uphill quenching cannot have a role in the post heat treatment processing of parts sensitive to the presence of residual stress.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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