Quench factor analysis of aluminium alloys using the Jominy end quench technique

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Determination of the time–temperature property C curve for aluminium alloys usually involves a large number of quenches and isothermal holds to calibrate a set of constants that describes the shape of the C curve for a particular property. The authors have used the Jominy end quench test to minimise the amount of work required for this type of analysis. By matching the Vickers hardness at regular intervals along the length of the Jominy test specimen with cooling curves generated using finite element analysis (FEA), the constants of the C curve equation were determined using a single Jominy test specimen. It was possible to successfully predict the hardness down to 65% of the maximum achievable hardness with a maximum error of only 2·4%.

Keywords: Quench factor analysis, 7000 series aluminium alloys, Joining end quench

Introduction

The Jominy end quench test has been extensively used to determine the hardenability of steels: ASTM 255. The test involves heating a standard cylindrical bar (25 mm in diameter and 100 mm in length) and then transferring it to a quenching fixture so that the specimen is held vertically 12·7 mm above an opening through which a column of water is directed against the bottom of the specimen. This results in a progressive decrease in the rate of cooling along the length of the bar. After the specimen has been a quenched, parallel flats are ground on the specimen surface and hardness measurements are taken along the length of the specimen.[1] While this test has seen widespread use in the steel industry there has been limited work conducted on aluminium alloys and other non-ferrous alloys. Early work by Bryant[2,3] investigated the quench sensitivity of 7000 series type alloys, while later investigations by 't Hart *et al.* used the Jominy test to study the effect of the cooling rate on the Vickers hardness, electrical conductivity, corrosion and microstructural properties of a number of high strength aluminium alloys.[4,5] More recent publications have promoted the use of the Jominy end quench test for aluminium alloys as a simple test that can provide information regarding quench sensitivity, microstructural characterisation and alloy development.[6] Newkirk *et al.* have used the Jominy end quench and quench factor analysis (QFA) to predict hardness and to demonstrate how process variables, such as delay time before aging and the ramp rates during aging can affect the final properties of the alloy.[7] However to date, the Jominy end quench technique has not been used to determine the range of constants necessary to construct a C curve.

History of quench factor analysis

Fink and Willey did much of the early work in describing the effects of the quench on the mechanical properties of aluminium alloys.[8] They used isothermal quenching techniques to construct C curves for the strength of 7075-T6 and corrosion resistance of 2024-T4. They predicted properties based on average cooling rate which provided satisfactory results if the cooling rate was uniform however problems arose if the cooling rate was non-uniform. Evancho and Staley improved upon the work by Fink and Willey so that properties could be predicted regardless of the shape of the cooling curve.[9–11]

Evancho and Staley described the TTP C curve by an equation of the form

$$C(T) = -k_1 k_2 \exp\left(\frac{k_3 k_4^2}{RT(k_4 - T)^2}\right) \exp\left(\frac{k_5}{RT}\right) \quad (1)$$

where $C(T)$ is the critical time required to precipitate a constant amount of solute (s), k_1 is the constant that equals the natural logarithm of the fraction untransformed during quenching, k_2 is the constant related to the reciprocal of the number of nucleation sites, k_3 is the constant related to the energy required to form a nucleus (J mol^{-1}), k_4 is the constant related to the solvus temperature (K), k_5 is the constant related to the activation energy for diffusion (J mol^{-1}), R is the gas constant (J mol^{-1} K^{-1}) and T is temperature (K).

To predict the mechanical property (hardness, strength, etc.) the following equation was used

$$\left(\frac{\sigma - \sigma_{\min}}{\sigma_{\max} - \sigma_{\min}}\right) = \exp(k_1 Q) \quad (2)$$

where Q is the quench factor, $\sigma_{\min}$ and $\sigma_{\max}$ are the

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minimum and maximum values respectively and σ is the predicted value.

Initially as $\sigma_{min} \ll \sigma_{max}$ in high strength alloys, Evancho and Staley let $\sigma_{min}=0$ to simplify the calculations. The quench factor can be determined from the following equation

$$Q = \int_{t_0}^{t_f} \frac{dt}{C(T)} \quad (3)$$

where t is time (s), t_0 is time at the start of the quench (s), t_f = quench finish time (s) and $C(T)$ is the critical time as a function of temperature; the loci of the critical times is the TTP C curve.

Using a large number of specimens, the cooling curves having been recorded during the quench, the k_2–k_5 constants are iteratively changed to minimise the difference between the predicted and measured properties.

The accuracy of this method was limited to the upper 10% of the strength of the alloy. Ives *et al.*[12,13] improved upon the model by assuming that σ_{min} was not equal to zero but as a temperature independent constant that was varied iteratively along with the k_2–k_5 values to minimise the error between the predicted and the measured properties. This improved the accuracy of the predictions to the upper 15% of the strength of the alloy. In many cases this level of accuracy was acceptable as many manufacturers are only interested in loses of up to 15%. However as the losses increased beyond 15%, the accuracy of the models described above decreased. Therefore a method was required that would be able to accurately predict the properties down to levels lower than were possible using the above techniques. The techniques described above fixed the value of σ_{min}, while the value actually varies with temperature. A new model was developed that could take this factor into account.[14]

The maximum strength of an aluminium alloy is achieved when quenched at an infinite rate from the solution heat treatment (SHT) temperature so that it retains the maximum amount of solute. If an alloy is quenched to a temperature below the SHT temperature, held isothermally until equilibrium is reached and then quenched, a proportionate amount of solute will be lost from the alloy. The strength after equilibrium has been reached σ_{min} is the maximum strength that can be developed if the material was solution heat treated at that temperature. As the isothermal holding temperature is decreased, more solute will be lost and σ_{min} decreases further. Because strength is proportional to the solute content, the σ_{min}–isothermal hold temperature relationship should follow the same trend as the solvus curve in an equilibrium phase diagram.

This improved QFA model developed by Staley and Tiryakioglu[14] assumes that the material loses an incremental amount of ability to develop the property $\Delta\sigma_j$ over each time interval Δt_j such that

$$\Delta\sigma_j = (\sigma_{j-1} - \sigma_{min(T_j)}) \left[1 - \exp\left(-\frac{\Delta t_j}{C(T)}\right)\right] \quad (4)$$

where $\Sigma_{j-1} + \Delta\sigma_j = \sigma_j$, $\Delta\sigma_j$ is the incremental amount of strength loss, Δt_j is the time interval (s), $\sigma_{min(T)}$ is the minimum strength and $\sigma_{min(T)}$ is a function of the equilibrium concentration at each temperature. For each subsequent incremental isothermal step, $\sigma_{j-1} = \sigma_{min(T)}$ is a function of the amount transformed during the previous incremental isothermal step.

σ at the end of the quench can then be found by subtracting the sum of the $\Delta\sigma_j$'s from σ_{max}

$$\sigma = \sigma_{max} - \sum_{j=1}^{j=n} \Delta\sigma_j \quad (5)$$

To successfully carry out QFA, three pieces of information are required.

(i) A time temperature property C curve for the alloy and temper in question.
(ii) The effect of isothermal holding temperatures on the ability of the alloy to develop that specific property (σ_{min}).
(iii) A cooling curve which will be used to predict the final property of the alloy.

A time temperature property C curve is usually constructed using isothermal holds. A range of temperatures is selected between the solution heat treatment temperature and the artificial aging temperature of the alloy. A number of specimens are quenched rapidly into a salt bath set at these temperatures and held for varying lengths of time and then quenched into cold water. The temperature of each specimen is recorded during the quench and the isothermal holds so that an accurate picture of the thermal history of the specimen is known. This is repeated for a large number of specimens. Using the cooling curves and the measured property, the constants of the C curve are then determined by QFA where values obtained from literature are initially used to predict the properties.[15] These initial k_2–k_5 constants from equation (1) are then altered iteratively so that the error between the predicted and the measured properties is minimised. Once values for the constants are known the properties at any location within a large component manufactured from that alloy can be accurately predicted if the cooling curves for that location are known. The amount of work required to determine the k_2–k_5 constants can be considerable as the cooling curves of each specimen in the isothermal holds needs to be recorded.

The authors used the Jominy end quench test to generate the large number of cooling curves required for the determination of the C curve.

The use of the Jominy test provides a rapid method of acquiring the required thermal data and Vickers hardness along the length of the specimen. Finite element analysis is used to determine the cooling curves at regular intervals along the length of the Jominy specimen.

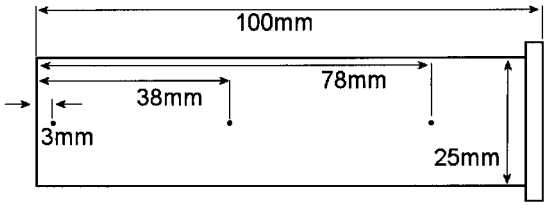
Experimental

Jominy end quench test

The Jominy end quench specimen was prepared in accordance with ASTM 255 from a 7175 rolled plate.

Figure 1 shows the Jominy specimen. 1·5 mm diameter holes were drilled at 3, 38 and 78 mm for type K thermocouples so that the temperature could be recorded during the quench.

The specimen was placed in an air recirculating furnace and allowed to soak for 2 h at 475°C. The specimen was then removed from the furnace and placed into the Jominy quench rig. Typical transfer time

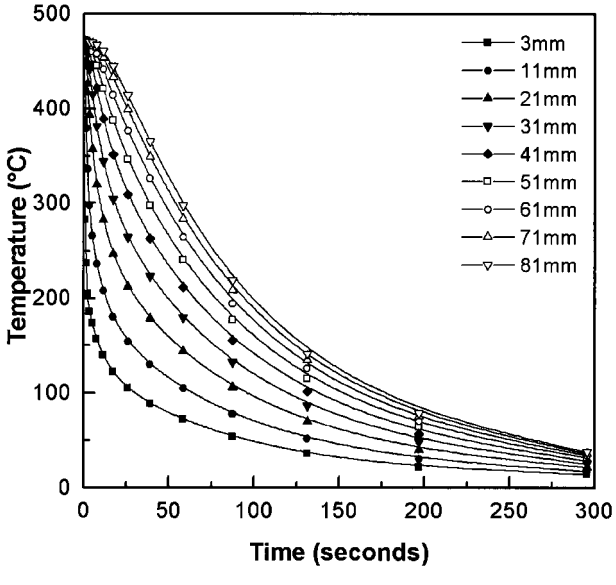


1 Jominy end quench specimen

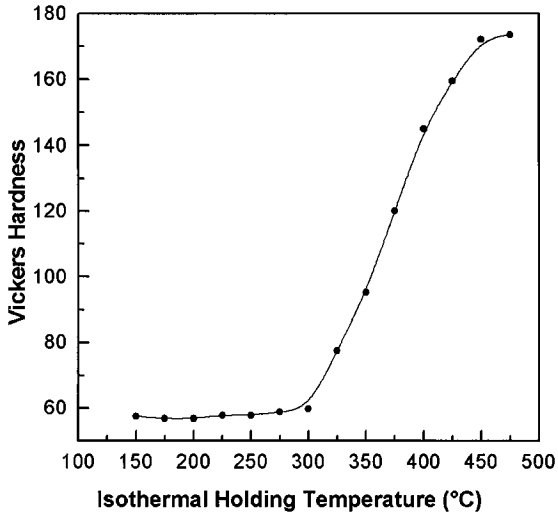
between the furnace door opening and the start of the quench was approximately 5 s. The specimen remained in the Jominy quench rig for 5 min to allow sufficient time to cool fully. It was then aged using a T73 type temper. The Jominy specimen was heated from room temperature to 170°C at 20 K h^{-1} and then held for 9 h before being allowed to air cool. Flats were machined on both sides of the specimen and the Vickers hardness was measured at 2 mm intervals along the length of the specimen. The Vickers hardness was measured using a Leco M-400-G1 microhardness tester using a 1 kg load. An average of three measurements was used for each location.

Cooling curve determination

In order to predict the cooling rates at every location along the length of the Jominy end quench specimen a heat transfer model of the Jominy end quench test piece was built using Abaqus. One quarter of the test piece was modelled because of symmetry using heat diffusion elements of type DC3D4 (four noded linear tetrahedron) for the head of the sample and DC3D8 (eight noded quadratic brick) elements for the main shaft of the sample. The use of different types of elements did not affect the final predictions as the area meshed with tetrahedron elements was small and was away from the main area of interest in the test piece. Properties for thermal conductivity, density and specific heat capacity were taken from the literature.[16] Cooling curves measured at 3 mm from the end of the Jominy end quench test piece were used as the main boundary condition to determine the rate of cooling of the remainder of the test piece. Radial heat transfer from the unquenched sides of the specimen was ignored as previous work has indicated that any heat transfer that may occur to the surrounding air has a minimal effect the hardness measured.[6]



2 Predicted cooling curves generated from the FEA Jominy end quench model representing distance from quenched end



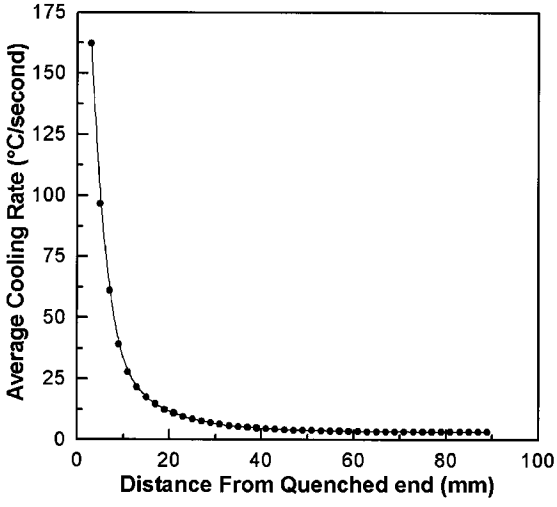
3 Effect of isothermal holding on Vickers hardness

The Jominy end quench test itself was repeated three times to determine cooling curves and therefore cooling rates for the locations indicated in Fig. 1. The quench was found to be repeatable from the cooling curves obtained. The measured cooling curves compared well with the finite element model predictions at 3, 38 and 78 mm from the quenched end. The cooling curves from selected distances from the quenched end of the Jominy can be seen in Fig. 2.

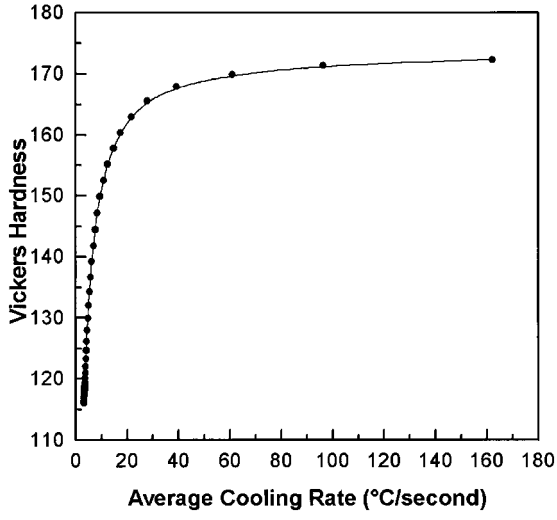
Effect of isothermal holding temperature on σ_{min} (hardness)

To determine the effect of the isothermal holding temperature on the Vickers hardness of 7175, the following procedure was used. Several small specimens of geometry $25 \times 25 \times 4$ mm were solution heat treated at a temperature of 475°C for a period of 2 h. One specimen was then removed from the furnace and rapidly quenched into room temperature water. The specimen was then transferred to a freezer set at a temperature of −22°C to retard any precipitation. The furnace temperature was then set to 25 K lower and held for a period of 24 h. It was assumed that the alloy had reached equilibrium conditions after this period of time. Another specimen was then removed and quenched into water and placed in the freezer. This process was repeated, decreasing the temperature in 25 K intervals until a temperature of 150°C was reached. The specimens were then aged using the T73 aging profile described previously and the Vickers hardness was measured. The relationship between Vickers hardness and the isothermal holding temperature for 7175 can be seen in Fig. 3.

A Boltzman sigmoidal equation was fitted to the curve to accurately determine the Vickers hardness at any temperature from 475°C to room temperature for use in the quench factor model.



4 Predicted average cooling rate between 400 and 250°C



6 Effect of cooling rate on the Vickers hardness of 7175

Discussion

Jominy end quench

The cooling rate varies considerably from one end of the Jominy to the other. Figure 4 shows the predicted average cooling rate between 400 and 250°C along the length of the Jominy specimen.

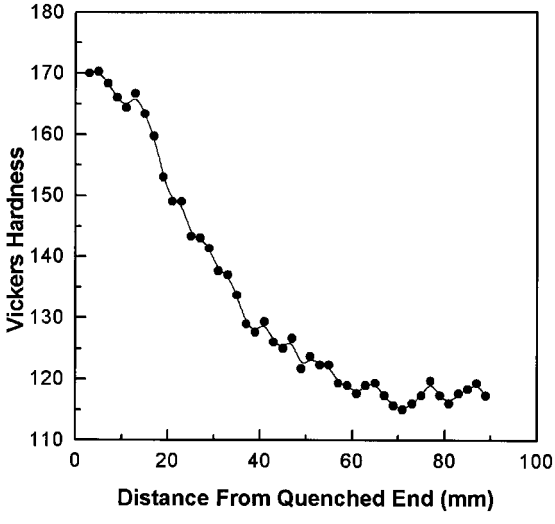
It can be seen that 3 mm from the quenched end of the specimen the cooling rate is rapid, recorded here at over 160 K s^{-1}. The cooling rate has decreased significantly to approximately 12 K s^{-1} 20 mm from the quenched end. Towards the end of the Jominy specimen the cooling rate decreases further to 3 K s^{-1}. Depending on the alloy, the cooling rate during the quench can have a significant effect its mechanical properties. Figure 5 shows the effect of the decreasing cooling rate on the Vickers hardness of 7175.

As the distance from the quenched end increases there is a steady reduction in the hardness of the alloy. It begins to level out at approximately 60 mm from the quenched end and maintains a hardness of close to HV120, roughly 65% of the maximum attainable hardness. Figure 6 shows the effect of the average cooling rate between 400 and 250°C on the Vickers hardness of 7175. There is not much effect on the hardness of 7175 with a cooling rate above 50 K s^{-1}; however, as the cooling rates decreases below 50 K s^{-1} there is a sharp decline in the hardness of the alloy.

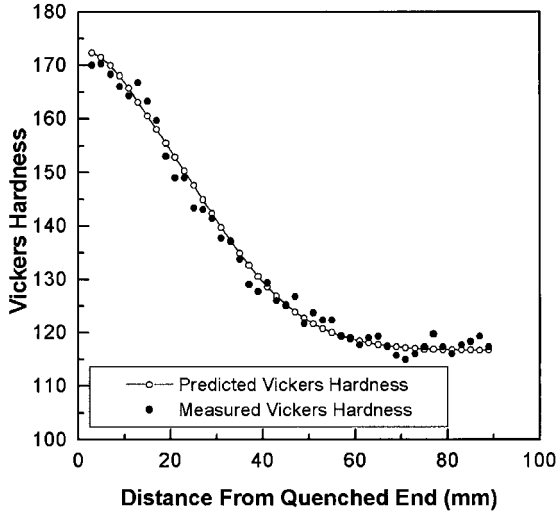
Quench factor analysis

A spreadsheet was set up using Microsoft Excel to enable QFA. All the cooling curves generated from the Abaqus model were imported into this spreadsheet. The spreadsheet enables the k_2–k_5 constants to be varied to minimise the misfit between the predicted and measured hardness values.

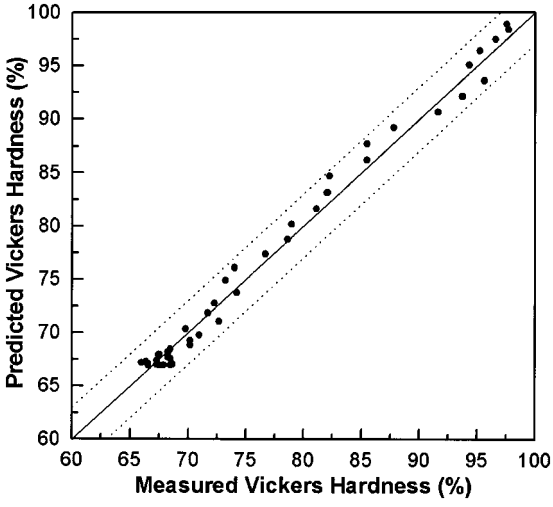
Values obtained from literature for the constants k_2–k_5 were used initially and varied iteratively until the mean squared error was minimised.[15] Recent work by Shuey *et al.* has suggested minimising the number of constants that are varied during the optimisation process to remove the instability of the Excel Solver.[17] Because the solvus temperature and the activation energy for diffusion are generally known, Shuey *et al.* proposed to fix these values while altering the remaining k_2 and k_3 constants. This removes a great amount of the processing time required when optimising the constants and generally makes the process more stable when using the Excel Solver.[17] Therefore for the purpose of this



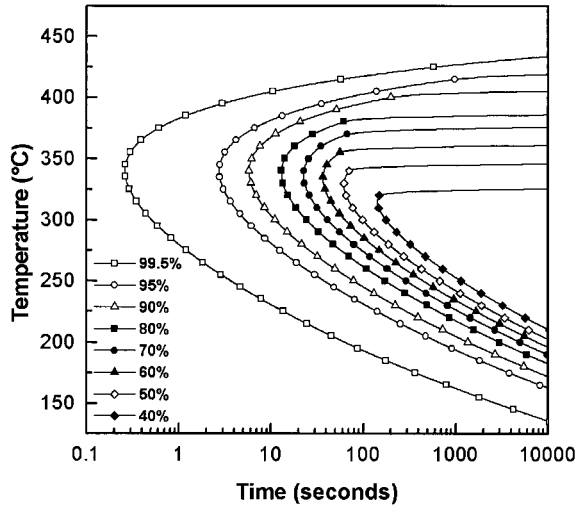
5 Vickers hardness of the Jominy end quench



7 Measured and predicted Vickers hardness



8 Relationship between measured and predicted Vickers hardness (percentage)



9 C curves representing iso-hardness curves for 7175–T73

paper, the k_4 value was fixed at 505°C (778 K) and k_5 (the activation energy for self diffusion) was fixed at 130 056 J mol^{-1} (Ref. 18). QFA works well when there is a large variation in the measured property, as this will ensure accuracy over a wide range of values.

Using the data generated from the Jominy end quench test, a total of 43 cooling curves were used to optimise the constants in equation (1). The results of the optimisation process can be seen in Figs. 7 and 8.

Figure 7 shows the measured hardness along the length of the Jominy specimen along with the predicted hardness curve after the constants in the C curve equation have been determined. Figure 8 shows the relationship between the measured and predicted hardness as a percentage of the total hardness. The dashed lines represent $\pm 3\%$ which is the error associated with using the Vickers hardness measurement technique.

From the results it is clear that the relationship between the measured and the predicted hardness is very good. The standard error between the measured and the predicted Vickers hardness is HV1·13 (0·65%) while the maximum difference generated between the measured and the predicted is HV4·1 (2·11%).

Figure 9 shows the C curve s that have been generated using the Jominy end quench test to calibrate the set of C curve constants. These represent iso-strength curves for 7175 aged to the T73 temper. Table 1 shows the k_2–k_4 constants that were used to construct the 7175 C curve.

As mentioned previously the amount of work required to generate C curves has been one of the stumbling blocks for the more widespread use of QFA. To generate the large number of cooling curves required for the accurate determination of the C curve constants, the cooling curves of a large number of specimens need to be determined during quenching and isothermal holding. By utilising the Jominy end quench test a large number of cooling curves can be generated from a single test specimen, thereby reducing the amount of work required to generate the C curve.

Table 1 Constants used in the construction of the 7175 C curve

Constants	k_2	k_3, J mol^{-1}	k_4, K	k_5, J mol^{-1}
7175-T73	7E-12	798	778	130 056

Conclusions

The Jominy end quench test is a quick and simple test that shows the effect of cooling rate on the hardness of an aluminium alloy. By utilising FEA to generate the cooling curves, it is possible to calibrate the constants of the C curve equation with less effort and much more rapidly than using isothermal holding data.

Further work will involve expanding the number of alloys and tempers, including electrical conductivity measurements, micro tensile testing and microstructural examinations.

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