Quench Factor Analysis of aluminium alloys using the Jominy End Quench technique

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Abstract
The Jominy End Quench has been used extensively to measure the hardenability of steels as it provides a method for studying a large number of different quenching conditions within a single test specimen, but has not been extensively used with non-ferrous alloys. Quench factor analysis of aluminium alloys usually involves recording the cooling curves of a large number of specimens as they are quenched into a salt bath set at various temperatures for varying lengths of time, to determine a range of constants for the time temperature property C-curve equation. This can be laborious and time consuming. To minimize the amount of effort and time required for Quench Factor Analysis, the authors applied the Jominy End Quench test to an aluminium 7000 series alloy and measured the Vickers hardness along the length of the specimen. Finite element analysis was used to accurately predict the cooling curves at regular intervals along the length of the Jominy specimen. Quench Factor Analysis was conducted and was found to accurately predict the Vickers hardness of the alloy with a standard error of 0.6%.

Keywords: Aluminium alloys, 7000, Quench Factor Analysis, Jominy End Quench

Introduction

The Jominy End Quench test has been extensively used to determine the hardenability of steels and has an associated ASTM standard: ASTM-255. The test involves heating a standard cylindrical bar (25.4mm in diameter and 102mm in length) to the proper austenitizing temperature and then transferring it to a quenching fixture so that the specimen is held vertically 12.7mm 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 180° apart are ground on the specimen surface and hardness measurements are taken along the length of the specimen. (Ref Steels Honeycombe). While this test has seen widespread use in the steel industry there was limited work conducted on aluminium alloys and other non-ferrous alloys. 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 several high strength aluminium alloys (ref x2). More recent publications have promoted the use of the Jominy End Quench test for the aluminium alloys as a simple test that can provide a wealth of information regarding quench sensitivity, microstructural characterization and alloy development (MacKenzie publications). Mackenzie also uses the Jominy end quench and quench factor analysis for predicting properties and how process variables, such as delay time before ageing and the ramp rates during ageing can affect the final properties of the alloy (ref).

Quench factor analysis has been used to predict the mechanical properties of heat treatable aluminium alloys for a number of years (ref). Some of the first work conducted was by Fink and Willey (ref), who developed Time-Temperature-Property C-curves for the 7075 in the late 1940's and used the average

cooling rate to try and predict the mechanical properties. They had limited success as their predictions were based on average cooling rate and did not take into account variations in the cooling rate during the quench. Evancho and Staley improved the model for predicting the mechanical properties.

The TTP C-curve can be described by an equation of the form:

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

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

To predict the mechanical property the following equation was used:

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

Where:
Q = Quench factor
σ_{min} = Minimum strength
σ_{max} = Maximum strength
σ = Predicted strength

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

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

t is time (s),
t_0 = 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.
Q = quench factor.

Three pieces of information are required to carry out quench factor analysis.

1. A time temperature property C-curve for the alloy and temper in question.
2. The effect of isothermal holding temperatures on the ability of the alloy to develop that specific property.
3. 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 ageing 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 the specimens 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. The constants of the C-curve are then determined by quench factor analysis where general values for the constants are initially used to predict the properties. These initial $k_2 - k_5$ constants are then altered iteratively so that the error between the predicted and the measured properties is minimized. Once values for the constants are known the properties at any location within a large component of the alloy can be accurately predicted if the cooling curves are known. **No work appears to have been done to accurately relate the position of the nose of the C-curve to the physical data recorded, just best fit will do.**
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. It has been shown that the isothermal holds can be used to accurately predict the properties based on continuous cooling curves. This paper proposes using continuous cooling curves to determine the constants of the C-curve equation. This means that the cooling curves that are generally observed during the quenching of large components are used to determine the constants for the C-curve equation. Quench factor analysis has been shown to be capable of accurately predicting properties based on a wide range of cooling rates provided reheating during the quench does not occur [ref].

The accuracy of this method was limited to the upper 10% of the strength of the alloy. Swartzendruber et al (ref) 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 minimize the error between the predicted and the measured properties. This improved the accuracy 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%. Problems arise when the loses are higher than those predicted by the techniques described above. Therefore a method was required that would be able to accurately predict the properties down to levels lower than were possible using the previous techniques. The techniques described above fixed the value of σ_{min}, while the value actually varies with temperature. Therefore a new model was required that could take this factor into account (ref).
The maximum strength of an aluminium alloy is achieved when quenched at an infinite rate from the solution temperature to retain the maximum amount of solute available for precipitation hardening. If an alloy is quenched to a temperature below the solution temperature, held isothermally until equilibrium is reached and subsequently quenched, a proportionate amount of solute will be lost from the alloy. Strength after equilibrium is 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. As the 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. The relationship between Vickers hardness and the isothermal holding temperature for 7175-T73 can be seen in Figure 1.

The newer Quench factor model 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:

$$\Delta\sigma_j = \left(\sigma_{j-1} - \sigma_{\min(Tj)}\right)\left[1 - \exp\left(-\frac{\Delta t_j}{Ct(T)}\right)\right]$$

Where:

$\Sigma_{j-1+}\Delta\sigma_j = \sigma_j$

$\Delta\sigma_j$ = Incremental amount of strength loss

Δt_j = Time interval

$C_t(T)$ = Critical time

$\sigma_{min(T)}$ = Minimum strength

$\sigma_{min(T)}$ is a function of the equilibrium concentration at each temperature at each incremental isothermal hold 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 i.e. σ_{j-1} is set equal to the predicated value of σ from the previous 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};

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

Experimental

Jominy End Quench Test

The Jominy End Quench specimen was prepared from 7175 rolled plate as per ASTM-255. The specimen was placed in a Carbolite air-recirculating furnace and allowed to soak for 2 hours. The specimen was then removed from the furnace and placed into the jominy quench rig. Typical transfer time between the furnace door opening and the start of the quench was approximately 5 seconds. The specimen remained in the jominy quench rig for 5 minutes to allow sufficient time to cool fully. The specimen was then aged to the T73 temper. Flats were machined on both sides of the specimen and the Vickers hardness was measured at 2mm intervals along the length of the specimen. The Vickers hardness was measured using a LECO M-400-G1 micro-hardness tester using a 1Kg load. An average of three measurements were used for each location. The change in the Vickers hardness along the length of the alloy can be seen in Figure 4.

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 modeled due to symmetry using heat diffusion elements of type DC3D4 (4-noded linear tetrahedron) for the head of the sample and DC3D8 (8-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 literature as previously descried [1]. Cooling curves measured at 3mm 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 does not affect the hardness measured [2].

The Jominy end quench test was repeated three times to determine cooling curves and hence cooling rates for the locations indicated in Figure 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 38mm and 78mm from the quenched end.

Effect of Isothermal holding temperature on σ_{min}

To determine the effect of the isothermal holding temperature on the Vickers hardness of 7175-T73, the following procedure was used. Several small specimens of geometry 25mm x 25mm x 4mm were solution heat-treated at a temperature of 475±2°C for a period of 1 hour. 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°C lower and held for a period of 24 hours. Another specimen was then removed and quenched into water and placed in the freezer. This process was repeated, decreasing the temperature in 25°C intervals until a temperature of 150°C was reached. The specimens were then aged to the T73 temper and the Vickers hardness was measured. The effect on the Vickers hardness with decreasing isothermal holding temperature can be seen in figure? The solvus temperature for 7175 is approximately 455°C so there will be no degradation in the properties of the alloy until the alloy is cooled to below this temperature. A minimum in the Vickers hardness is reached at a temperature of close to 300°C. An equation was fitted to the curve to accurately determine the Vickers hardness at any temperature from 475°C to room temperature.

Identification of the temperature of the nose of the C-curve

It is necessary to determine at what temperature the nose of the C-curve occurs at so that the shape of the C-curve is as accurate as possible. When iteratively changing the k_2 and k_3 constants it is possible to get the best fit to the data possible but the shape of the C-curve will not accurately reflect the true position of the C-curve. To determine the temperature at which the nose of the C-curve is located a small number of isothermal holds are conducted at temperatures of 270°C, 310°C, 350°C and 390°C. A single time period is used, in this case a hold of 10 seconds. For each hold there is a loss in the hardness of the alloy, the loss increasing the closer you get to the nose of the C-curve. The temperatures selected should give a wide enough range so as to be above and below the temperature of the nose of the C-curve. Plotting the hardness versus temperature shows the temperature at which precipitation rates are fastest, hence the location of the nose of the C-curve. This is another variable that can be included into the iterative process to determine the k_2 and k_3 constants so that the most accurate fit to the data can be determined while still ensuring that the shape of the C-curve is as accurate as possible.

Results and Discussion

Quench Factor Analysis

Candidate values for the constants $k_2 – k_5$ that were used initially were varied iteratively until the mean squared error was minimized. Recent work by Tiriacioglu has suggested minimizing the number of constants that are varied during the optimization process to remove the instability of the Excel Solver. Since the solvus temperature and the activation energy for diffusion are generally known, they proposed to fix these values while altering the remaining k_2 and k_3 constants. This removes a lot of processing time required when optimizing the constants and generally makes the process more stable when using the Excel Solver (ref). Therefore for the purpose of this paper, the k_4 value was fixed at

455°C (728K), the solvus temperature confirmed by DSC analysis. The activation energy for self diffusion was fixed at 133592 J/mol (ref).

Figure 3 shows the average cooling rate between 400°C and 250°C along the length of the Jominy specimen. It can be seen that 3mm from the quenched end of the specimen the cooling rate is rapid, recorded here at over 160°C per second. 20mm from the quenched end the cooling rate has decreased significantly to approximately 12°C/second. Towards the end of the Jominy specimen the cooling rate decreases further to 3°C/second. Depending on the alloy, the cooling rate during the quench can have a significant effect on the mechanical properties of the alloy. Figure 4 shows the effect of the decreasing cooling rate of the Vickers hardness of 7175-T73. From the results presented here, 7175 is a quench sensitive alloy with the cooling rate having a massive effect on the hardness of the alloy. The hardness does not decrease much within the first 15mm of the quenched end of the Jominy specimen but beyond this point there is a rapid reduction in the hardness of the alloy. The hardness begins to level out at approximately 60mm from the quenched end and maintains a hardness of close to 120 HV, 65% of the maximum attainable hardness. Figure 7 shows the effect of the average cooling rate between 400°C and 250°C on the Vickers hardness of 7175-T73. There is not much effect on the hardness of 7175 with a cooling rate above 50°C/second, however as the cooling rates decrease below 50°C/second there is a sharp decline in the hardness of the alloy.
Using the data generated from the Jominy End Quench test, a total of 44 cooling curves were used to optimize the constants in equation ? The results of the optimization process can be seen in Figure 5 and Figure 6. Figure 5 shows the measured hardness along the length of the Jominy specimen along with the predicted hardness after the constants in the C-curve equation have been determined. Figure 6 shows the relation of the measured and predicted hardness as a percentage of the total hardness. The dashed lines represent ±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 excellent. Standard error between the measured and the predicted Vickers hardness is 1HV (0.6%) while the maximum difference generated between the measured and the predicted is 4.6 HV (2.65%).
Figure 8 shows the C-curves 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-T73. Quench sensitive alloys generally have the nose of their C-curves at very short times, indicating that very little time is required for precipitation to occur. For alloys that are not quench sensitive the nose of the curve will be shifted further to the right. The temperature at which the nose of the C-curve is located can also be used as an indication of the quench sensitivity of an alloy. If two alloys have the nose of the C-curve located at the same time but at different temperatures then the alloy with the nose of the C-curve located at the higher temperature should be less quench sensitive than the alloy with the C-curve located at the lower temperature. Since the most rapid part of the quench is typically at the start of the quench, less time will be spent in the precipitation zone of the alloy with the nose of the C-curve at the higher temperature than the alloy with the nose of the C-curve at the lower temperature.

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 quench factor analysis. 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 needs to be determined during quenching and isothermal holding. By utilizing 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 a C-curve. This procedure will

work equally well for electrical conductivity but has not been examined in this paper. There is a close relationship between the hardness of an alloy and the tensile strength of an alloy however it is unwise to use the hardness C-curve constants for tensile properties (ref) although work has been conducted to accurately relate the Vickers hardness of an alloy to the tensile strength for a number of alloys (ref).
Work is also being conducted (ref) to try and relate the strength of the alloy to the solvus curve in the equilibrium phase diagram and has had some success. The approach used in this paper to allow the alloy to reach equilibrium while holding at a specific temperature appears to work quite well in enabling the accurate prediction of the hardness of the alloy. While this approach takes time, the actual amount of work required is very small.

Conclusions and Future work

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 utilizing the continuous cooling curves generated during the quench to calibrate the constants of the C-curve equation, it is possible to construct a Vickers Hardness C-curve 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 and microstructural examinations.

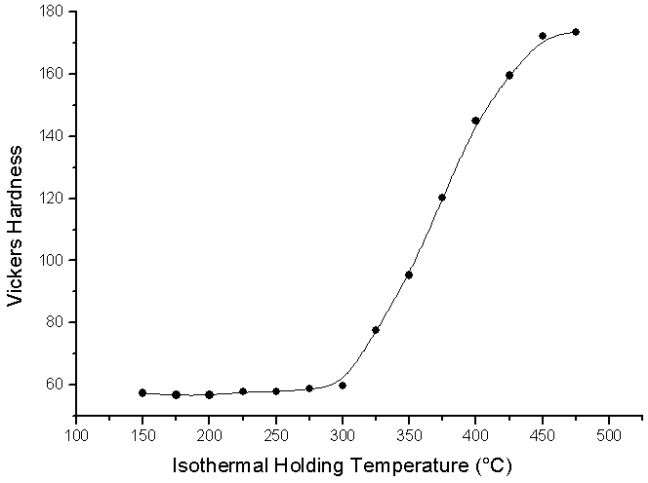

Figure 1 Effect of isothermal holding on Vickers hardness

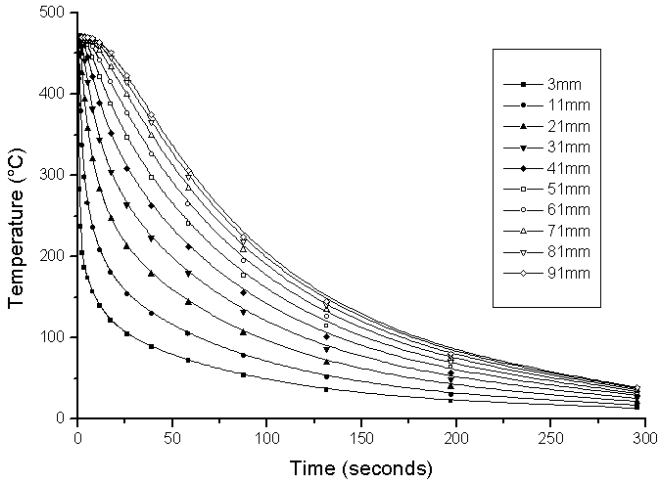

Figure 2 Cooling curves generated from the Jominy End Quench representing distance from quenched end

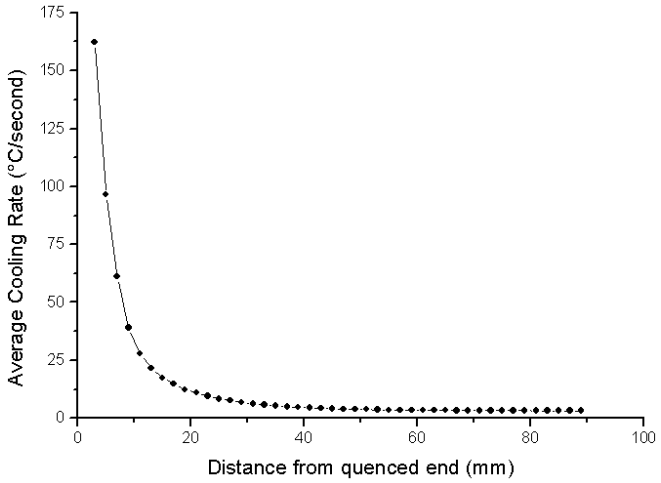

Figure 3 Average cooling rate between 400°C and 250°C

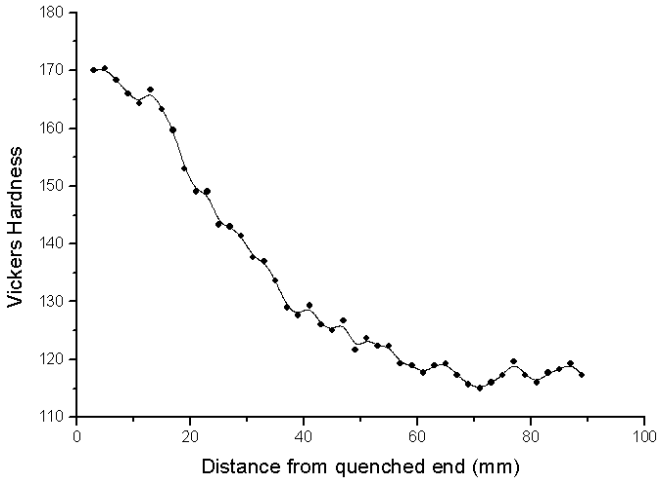

Figure 4 Vickers hardness of the Jominy End Quench

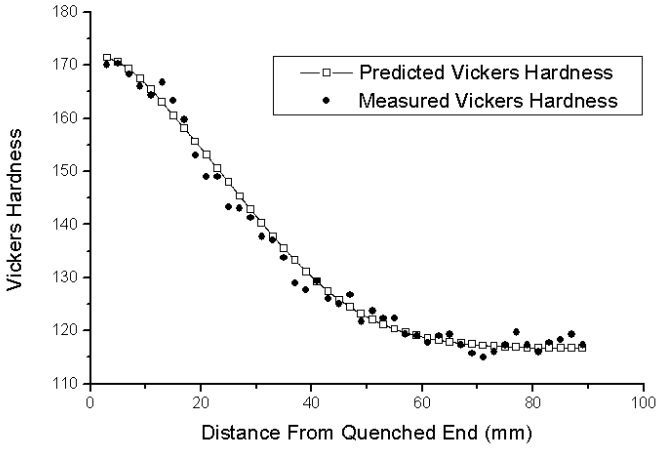

Figure 5 Measured and predicted Vickers hardness

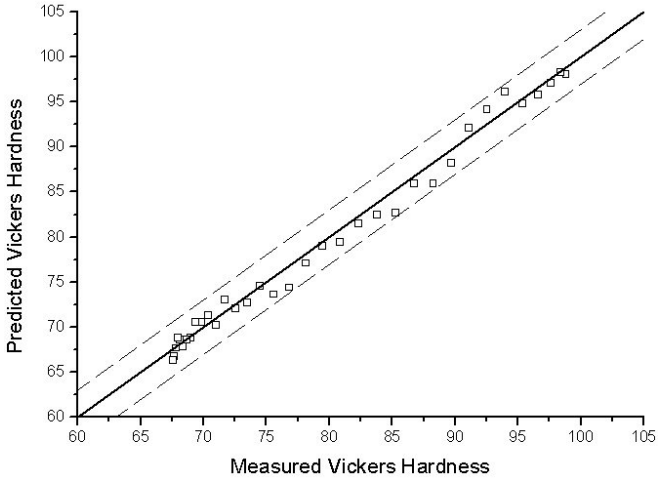

Figure 6 Relationship between measured and predicted Vickers hardness (percentage)

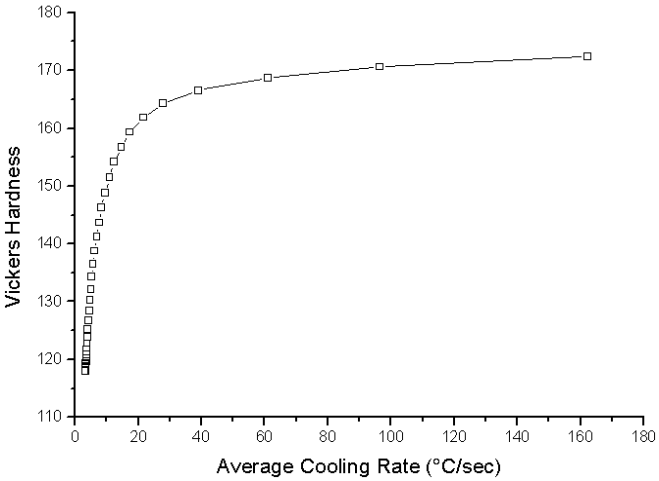

Figure 7 Effect of cooling rate on the Vickers hardness of 7175-T73

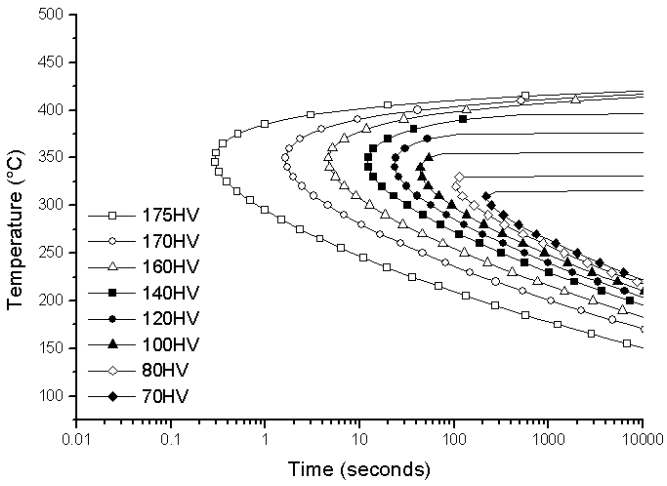

Figure 8 C-curves representing iso-hardness curves for 7175-T73