Retrogression and Reaging of AA7075 and AA6013 Aluminum Alloys

KATHERINE E. RADER, JON T. CARTER, LOUIS G. HECTOR Jr., and ERIC M. TALEFF

Retrogression and reaging (RRA) is of interest to the automotive industry for manufacturing components of high-strength aluminum alloys. RRA heat treatments are investigated for AA7075-T6 and AA6013-T6 materials. Retrogression is demonstrated to be a thermally activated process reasonably characterized with a single activation energy. Activation energies for retrogression are measured as 97 ± 7 and 160 ± 30 kJ/mol for AA7075-T6 and AA6013-T6, respectively. Critical retrogression times, t_R* and t_R^{max}, are defined and measured across a range of retrogression temperatures. These data are used with the concept of reduced time to predict combinations of temperature and time that produce successful retrogression heat treatments. Recommended retrogression heat treatments are 200 °C for 3 to 12 minutes for AA7075-T6 and 240 °C for 7 minutes for AA6013-T6. Data from reaging heat treatments confirm a significant RRA response in AA6013. Recommended reaging heat treatments are 120 °C for 24 hours for retrogressed AA7075 and 190 °C for 1 hour for retrogressed AA6013. A reaging heat treatment that simulates the automotive paint-bake cycle, 185 °C for 25 minutes, is almost as effective as the recommended reaging heat treatment for AA6013 but is significantly less effective than the recommended reaging heat treatment for AA7075.

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I. INTRODUCTION

RETROGRESSION and reaging (RRA) is a two-step heat treatment that is useful in manufacturing components from precipitation-strengthened aluminum alloys. RRA was introduced by Cina and Ranish to improve the stress-corrosion-cracking resistance of AA7075 aluminum.[1,2] Appropriate RRA heat treatments of aluminum alloy AA7075 in the T6 temper retain or slightly increase strength and improve stress-corrosion-cracking resistance to approximately that of the T7 temper.[2] The first step of the RRA process is retrogression. During retrogression, an alloy in the T6 temper is soaked at a temperature between its aging and solutionizing temperatures and is then rapidly cooled.[1,2] During the retrogression heat treatment, strength decreases. Cina and Ranish recommended a retrogression heat treatment of 240 °C for 12 seconds for AA7075,[1,2] but subsequent investigators suggested alternative times and temperatures.[3–7] The second step of the RRA process is reaging, which recovers strength lost during retrogression. For AA7075, Cina and Ranish recommended a reaging heat treatment of 121 °C for 48 hours,[2] but subsequent investigators applied the more typical T6 aging heat treatment of 120 °C for 24 hours to reaging.[5,7]

The effects of RRA on the room-temperature tensile strength of AA7075-T6 are shown schematically in Figure 1. The time at which the local minimum in strength occurs during retrogression is defined as the first critical retrogression time, t_R*. The time at which the local maximum in strength occurs during retrogression is defined as the second critical retrogression time, t_R^{max}. Park and Ardell investigated changes of the precipitate structures in AA7075-T6 throughout RRA.[8] During the retrogression heat treatment, four reactions occur: (1) the dissolution of small η' precipitates, (2) the transformation of large η' precipitates into η precipitates, (3) the nucleation of η precipitates, and (4) the growth of η precipitates.[8] The initial loss of strength during the retrogression heat treatment up to t_R*, shown in Figure 1, is dominated by the dissolution of the strengthening η' precipitates.[8] The slight recovery of strength from t_R* to t_R^{max} is from the precipitation and growth of η precipitates.[8] Continued coarsening of η precipitates beyond t_R^{max} produces a loss of strength.[8] If AA7075 is retrogressed for a time less than t_R^{max}, the η' precipitates only partially dissolve, and the η precipitates

KATHERINE E. RADER and ERIC M. TALEFF are with the Department of Mechanical Engineering, The University of Texas at Austin, Austin, TX 78712. Contact e-mail: Kate.Rader@utexas.edu JON T. CARTER and LOUIS G. HECTOR JR. are with Global Research and Development, General Motors, Warren, MI 48092.

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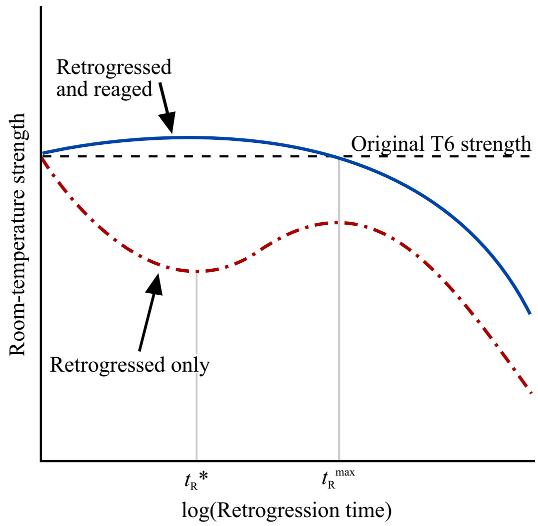

Fig. 1—This schematic demonstrates how room-temperature tensile strength changes with retrogression time for AA7075 sheet material subjected to retrogression alone and for material reaged after retrogression. The original T6 strength is represented by the dashed line. The critical retrogression times indicated are t_R* and t_R^{max}.

retain a relatively fine size. The original strength of the alloy can then be recovered with a single reaging heat treatment, which re-precipitates and grows η' precipitates.[8] The retrogression time recommended for the best strength from RRA heat treatments is generally equal to t_R*.[2–8] If the alloy is retrogressed for a time longer than t_R^{max}, the original peak-aged strength cannot be recovered through reaging because the necessary solute elements are consumed by coarsening precipitates, preventing the formation of fine η' and η precipitates.[8] During retrogression up to t_R^{max}, grain boundary precipitates coarsen. Retrogression for a time approximately equal to t_R^{max}, followed by reaging, produces stress-corrosion-cracking resistance similar to the T7 temper.[2–4,6,8–10] This improvement in stress-corrosion-cracking resistance was attributed by different investigators to coarsened grain boundary precipitates,[6,8] improved thermodynamic stability of the precipitate structure,[9] and changes to dislocation structures near grain boundaries.[10]

Existing applications and potential future applications of RRA extend beyond merely improving the stress-corrosion-cracking resistance of AA7075.[1,2,11,12] Ivanoff *et al*. demonstrated that the ductility of AA7075 during retrogression at a temperature of 200 °C is approximately double that at room temperature, which opens up new possibilities for warm forming.[7] The strength of the heat-affected zone in welded AA7xxx-series aluminum alloys is reported to be improved by a RRA heat treatment.[13] RRA has been applied to AA6xxx-series aluminum alloys to improve manufacturability during deformation processing. Retrogression heat treatments are used for compression-fit joining of AA6005, AA6061, and AA6063.[14–16] One method of producing ladders from AA6061 is to locally retrogress extruded tube sections to improve ductility prior to room-temperature forming.[16,17] Retrogression heat treatments improve the room-temperature trimming, flanging, and springback of AA6111.[18] Despite these practical applications of RRA, relatively little data on RRA of AA6xxx-series aluminum alloys are available in the literature.[13–18]

The automotive industry is interested in high-strength aluminum alloys for vehicle light-weighting,[7,18–26] and RRA heat treatments are potentially useful for manufacturing components of these materials. AA7075-T6 is of interest for its high strength, but its susceptibility to stress corrosion cracking is a concern.[2,7,19–24,27] The high-strength aluminum alloy AA6013-T6 is of interest as a lower cost option that is less susceptible to stress corrosion cracking.[28] The only RRA data available for AA6013 are from preliminary work by the present authors.[29] The conditions required for reaging are of particular interest to automobile manufacturers in relation to the automotive paint-bake process. The paint-bake process involves heat treatments used to cure paint applied to the vehicle body-in-white.[7,13,30,31] Ideally, the paint-bake process might be used as a reaging heat treatment for retrogressed materials in the body-in-white. One recommended reaging heat treatment for AA7075 is 120 °C for 24 hours, which is the same as the alloy's T6 aging heat treatment.[5,7,32,33] This reaging temperature is well below temperatures typical of the paint-bake process, which range from 170 to 185 °C.[7,13,30,31] The T6 aging heat treatment for AA6013 is 190 °C for 4 hours.[28] If AA6013 exhibits a RRA response and if its aging temperature is also an effective reaging temperature, then the paint-bake process may be useful for reaging AA6013.

The concept of reduced retrogression time is a useful means to design and compare retrogression heat treatments. The critical retrogression times, t_R* and t_R^{max}, depend on retrogression temperature. As retrogression temperature increases, these critical retrogression times decrease.[2–7] Ivanoff *et al*. hypothesized that retrogression is a thermally activated process that can be described by a single activation energy.[7] They proposed the concept of reduced retrogression time, τ_R, which uses an Arrhenius relationship to relate retrogression time and retrogression temperature.[7,34] Reduced retrogression time is defined as

$$\tau_R = t_R \times \exp\left(-\frac{Q_R}{RT_R}\right), \qquad [1]$$

where t_R is retrogression time, Q_R is the activation energy of retrogression, R is the universal gas constant, and T_R is retrogression temperature in Kelvin.[7] Ivanoff *et al*.,[7] followed by Rader *et al*.,[35] demonstrated that critical retrogression times measured at different retrogression temperatures align to a single reduced retrogression time, allowing the prediction of critical retrogression times across a range of intermediate retrogression temperatures. Equation [1] is particularly useful if it can effectively predict combinations of retrogression times and temperatures suitable to manufacturing operations, which may impose practical constraints on the times and/or temperatures that can be used.

The present investigation tests the hypothesis of Ivanoff *et al*. that retrogression is a thermally activated process that can be characterized with a single activation energy by measuring that activation energy independently of mechanical tests. Both AA7075 and AA6013 sheet materials are studied. Differential scanning calorimetry (DSC) is used to measure the activation energy for retrogression, Q_R, of each material. The present study investigates whether AA6013-T6 exhibits a significant RRA response. The room-temperature hardnesses of AA7075 and AA6013 are measured after retrogression heat treatments to determine the critical retrogression times at several temperatures. The activation energies measured for retrogression are used to calculate critical reduced retrogression times. These reduced retrogression times are used to predict retrogression behaviors and produce recommendations for retrogression heat treatments. The responses of retrogressed AA7075 and AA6013 to a variety of reaging heat treatments, including a simulated paint-bake heat treatment, are measured. Recommendations are made for reaging times and temperatures. The nomenclature used in this study is listed in Table I.

Table I. Nomenclature

Acronym	Description
DSC	differential scanning calorimetry
HF	instantaneous heat flow
HF_{max}	maximum heat flow within the interval of measurement
HF_{min}	minimum heat flow within the interval of measurement
HF_P	peak heat flow
HF_{rel}	relative heat flow
Q_R	activation energy for retrogression
R	retrogressed condition
$RMSE$	root-mean-square-error
RPB	retrogressed-and-paint-baked condition
RRA	retrogressed-and-reaged condition
T_P	peak temperature
T_R	retrogression temperature
T_{RA}	reaging temperature
t_R	retrogression time
t_R*	first critical retrogression time
t_R^{max}	second critical retrogression time
t_{RA}	reaging time
τ_R	reduced retrogression time
τ_R*	first critical reduced retrogression time
τ_R^{max}	second critical reduced retrogression time

II. EXPERIMENTAL PROCEDURES

Two commercial aluminum alloy sheet materials are studied: AA7075 (Al-Zn-Mg-Cu) and AA6013 (Al-Mg-Si-Cu). The nominal compositions of these alloys are listed in Table II, according to the respective producers.[28,32] The AA7075 sheet material was received in the T6 temper, and the AA6013 sheet material was received in the T4 temper. Both sheet materials have a 2 mm thickness as received.

Cylindrical specimens measuring 4 mm in diameter were blanked from the AA7075-T6 and AA6013-T4 sheets for differential scanning calorimetry (DSC) experiments. These specimens were first ground flat to a thickness of approximately 1.5 mm with SiC papers to a final grit of 600.[36,37] The specimens were then solutionized and subsequently aged to T6 tempers. Specimens of AA7075 were solutionized at 480 °C for 1 hour in a tube furnace and immediately water-quenched upon removal. These specimens were then aged to the T6 temper at 120 °C for 24 hours in a tube furnace and immediately water-quenched.[27,32,33] Specimens of AA6013 were solutionized at 570 °C for 1 hour in a tube furnace and immediately water-quenched. These specimens were then aged to the T6 temper at 190 °C for 4 hours in a tube furnace and immediately water-quenched.[28] The T6 condition was confirmed for each material after heat treating through Vickers hardness testing[38] of a few selected specimens. For AA7075, the published Vickers hardness of the T6 temper was referenced.[39] For AA6013, the published tensile strength of the T6 temper was converted to Vickers hardness for reference.[28,39–41] For each material, the specimens used for DSC experiments were taken from the same manufacturing lot and heat treatment batch.

During the DSC experiments, specimens were heated at a constant rate from 25 °C up to 300 °C for AA7075 or up to 350 °C for AA6013. Specimens of AA7075-T6 were tested using a Mettler™ Thermogravimetric Analyzer, Model TGA/DSC 1. Five different heating rates were applied using this instrument: 5, 10, 15, 20, and 25 °C/min. Three experiments were conducted at each heating rate. Specimens of AA6013-T6 were tested using a NETZCH™ Pegasus DSC 404 F1, which provided a sensitivity sufficient to resolve heat flow changes in the AA6013-T6 specimens. Seven heating rates were applied using this instrument: 2.5, 3.5, 5, 8, 10, 12, and 15 °C/min. One experiment was conducted at each heating rate. Heat flow normalized by specimen mass was measured as a function of specimen temperature for each experiment.

To study the effects of retrogression heat treatments on room-temperature hardness, 2-mm-thick sheets of AA7075-T6 and AA6013-T4 were sheared into specimens measuring 25 × 25 × 2 mm. Each specimen was ground flat using SiC papers to a final grit of 600 [36,37] and engraved with a unique specimen identifier. Specimens of AA7075 were retained in the as-received T6 temper. Specimens of AA6013, received in the T4 temper, were solutionized at 570 °C for 1 hour, water-quenched, and then aged at 190 °C for 4 hours to produce the T6 temper.[28] Three hardness measurements

Table II. The Nominal Compositions of AA7075 and AA6013 in Weight Percent[28,32]

Alloy	Zn	Mg	Cu	Cr	Fe	Si	Mn	Ti	Al
AA7075	5.1 to 6.1	2.1 to 2.9	1.2 to 2.0	0.18 to 0.28	< 0.5	< 0.4	< 0.3	< 0.2	bal.
AA6013	≤ 0.25	0.8 to 1.2	0.6 to 1.1	≤ 0.1	≤ 0.5	0.6 to 1.0	0.2 to 0.8	≤ 0.1	bal.

Table III. Retrogression Temperatures, T_R, and Times, t_R

Alloy	T_R (± 2 °C)	Shortest t_R (s)	Longest t_R (s)
AA7075-T6	192	30	5400
	200	25	2880
	210	20	1350
	220	15	600
AA6013-T6	230	10	6310
	240	10	1580
	250	10	1000
	270	10	3600
	285	10	600

Table IV. Reaging Temperatures, T_{RA}, and Times, t_{RA}

Alloy	T_{RA} (± 2 °C)	t_{RA} (min)
AA7075	185	25
	120	1440
AA6013	185	25
	190	60
		120
		180
		240

were taken from each specimen in the T6 condition using an automated Rockwell B hardness tester to calculate a mean hardness baseline for each T6 condition.[42] A 1/16 in diameter steel ball hardness indenter was used for all Rockwell B hardness tests (HRBS).[42] All specimens were retrogressed from the T6 temper in a molten salt bath. The retrogression temperatures and ranges of retrogression times studied for each material are listed in Table III. The temperature of the salt bath was measured with a K-type thermocouple and kept within ± 2 °C of the desired temperature. The specimens were removed and immediately water-quenched at the end of each retrogression heat treatment. Retrogression heat treatment times were controlled to within one second. The room-temperature hardness of each specimen was measured within a few hours of retrogression using an automated Rockwell B hardness tester.[42] Five hardness measurements were made for each retrogressed specimen.

After retrogression, specimens were reaged to study the effects of reaging heat treatments on room-temperature hardness. These reaging heat treatments are listed by alloy in Table IV. Specimens of retrogressed AA7075 were reaged in a box furnace; specimens of retrogressed AA6013 were reaged in a convection furnace. Specimen temperature was monitored with a K-type thermocouple and was controlled to within ± 2 °C of the desired reaging temperature. The reaging heat treatment times were controlled to within one minute. Upon completion of each reaging heat treatment, specimens were removed from the furnace and immediately water-quenched. Five hardness measurements were made for each specimen within 12 hours of the reaging heat treatment using an automated Rockwell B hardness tester.[42]

Two reaging heat treatments were studied for AA7075. The first, 185 °C for 25 minutes, simulates a paint-bake process for automotive manufacturing.[7,13,30,31] The second, 120 °C for 24 hours, is a recommended reaging heat treatment for AA7075.[5,7] All of the retrogressed specimens of AA7075 were subjected to one of these two reaging heat treatments. Five reaging heat treatments were studied for AA6013. Specimens of AA6013, representing all retrogression times studied for retrogression temperatures of 230, 240, and 250 °C, see Table III, were reaged with a simulated paint-bake heat treatment of 185 °C for 25 minutes.[7,13,30,31] Specimens representing retrogression times of 100, 400, and 1000 seconds at a retrogression temperature of 240 °C were reaged at 190 °C for 1, 2, 3, and 4 hours. The 190 °C temperature was chosen because it is the standard T6 aging temperature for AA6013.[28]

III. RESULTS

A. *Differential Scanning Calorimetry*

Examples of DSC data are shown in Figure 2 with endothermic peaks upward. The data are plotted as relative heat flow to facilitate comparisons across different heating rates. Relative heat flow, HF_{rel}, is defined as

$$HF_{\mathrm{rel}} = 1 - \frac{HF_{\max} - HF}{HF_{\max} - HF_{\min}}, \qquad [2]$$

where HF is the instantaneous measured heat flow, and $HF_{\max}$ and $HF_{\min}$ are the maximum and minimum heat flows within the interval of measurement. The endothermic peaks illustrated in Figure 2 are associated with the dissolution of precipitates.[8,43,44] For AA7075, these precipitates are η'.[8,43] For AA6013, these precipitates are β'' and Q'.[44–46] As the heating rate increases, the temperature of the endothermic peak, T_P, also increases. To measure the peak temperature, a third-degree polynomial was fit to HF as a function of temperature in the vicinity of the peak.[47] The maximum of the polynomial was calculated to determine the heat flow, HF_P, and temperature, T_P, at the peak. The mean peak temperature for each test condition is reported in Table V.

Estimated uncertainties in T_P are also reported. The uncertainty of each T_P measurement was estimated by first calculating the root-mean-square-error ($RMSE$) in HF of the polynomial. The span of temperatures about T_P encompassing $(HF_P - RMSE) \leq HF \leq HF_P$ was taken as an estimate of the uncertainty in T_P. Appropriate propagation of error was used to estimate the uncertainty of each mean peak temperature where multiple experiments were conducted.[48]

B. *Retrogression Heat Treatments*

Room-temperature hardness (HRBS) data measured after retrogression are shown in Figure 3. Hardness is used as a surrogate for tensile strength. All specimens were in a T6 temper condition prior to retrogression. The mean hardness of AA7075-T6 specimens prior to retrogression is 90.1 ± 0.4 HRBS. The mean hardness of AA6013-T6 specimens prior to retrogression is 67.8 ± 1.4 HRBS. The uncertainty of each mean hardness is defined as the sample estimate of standard deviation. The shape of the hardness curves for AA7075 matches those in literature, see Figure 1.[2–8] As specimens are retrogressed for increasingly longer times, there is an initial decrease in hardness, followed by a slight increase in hardness. This produces a local minimum of hardness at $t_R{}^*$. After the slight increase in hardness, there is a second decrease in hardness, producing a local maximum of hardness at t_R^{max}. The region from $t_R{}^*$ to t_R^{max} is shaded as gray in Figure 3(a). As the retrogression temperature, T_R, increases, the times at which $t_R{}^*$ and t_R^{max} occur generally decrease. This is represented in Figure 3(a) by the shaded region narrowing and shifting to the left as retrogression temperature increases. The times $t_R{}^*$ and t_R^{max} are listed in Table VI. These critical times are longer than those reported by Cina and Ranish but are similar to those reported by Rajan *et al.* and by Park.[2,4,6] The T6 aging heat treatment for AA7075 can range from 24 to 48 hours in duration.[2–4,27,32,33] Cina and Ranish used a T6 aging heat treatment of 121 °C for 48 hours.[2] Both Rajan *et al.* and Park used much shorter T6 aging heat treatments of 120 °C for 29[4] and 24 hours,[6] respectively; the latter is the same aging time used in the present study. This difference in aging heat treatments is one possible source of the small $t_R{}^*$ and t_R^{max} values reported by Cina and Ranish compared to other studies.

The shape of the hardness curves for AA6013, shown in Figure 3(b), is different than that of AA7075, see Figures 1 and 3(a). Room-temperature hardness initially decreases and then plateaus as retrogression time increases. After the plateau, hardness further decreases as retrogression time increases. The beginning and end times of the plateau are defined as $t_R{}^*$ and t_R^{max}, respectively. The region of the plateau (*i.e.*, the region from $t_R{}^*$ to t_R^{max}) is shaded as gray in Figure 3(b). The times $t_R{}^*$ and t_R^{max} measured for each retrogression

Table V. Temperature, T_P, at Which the Endothermic Peak Occurs

Alloy	Heating Rate (°C/min)	T_P (°C)
AA7075-T6	5.0	200 ± 5
	10.0	211 ± 2
	15.0	218 ± 1
	20.0	224 ± 1
	25.0	230 ± 1
AA6013-T6	2.5	226 ± 4
	3.5	233 ± 6
	5.0	234 ± 4
	8.0	240 ± 3
	10.0	243 ± 3
	12.0	246 ± 2
	15.0	251 ± 2

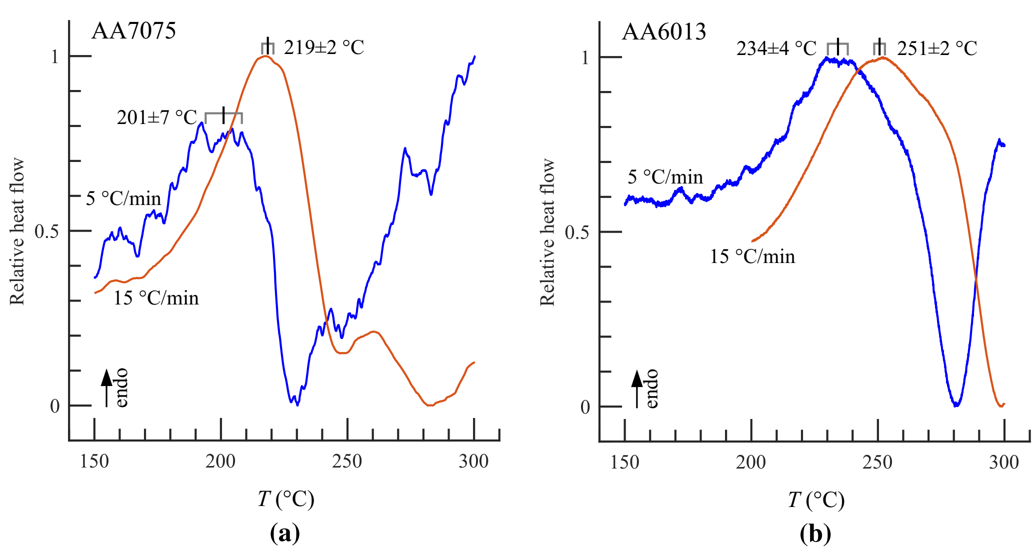

Fig. 2—DSC data for relative heat flow, see Eq. [2], are plotted as endotherms upward *vs* temperature for (*a*) AA7075-T6 and (*b*) AA6013-T6 for two heating rates, 5 and 15 °C/min. The temperatures of endothermic peaks associated with retrogression are identified by annotations in the figures.

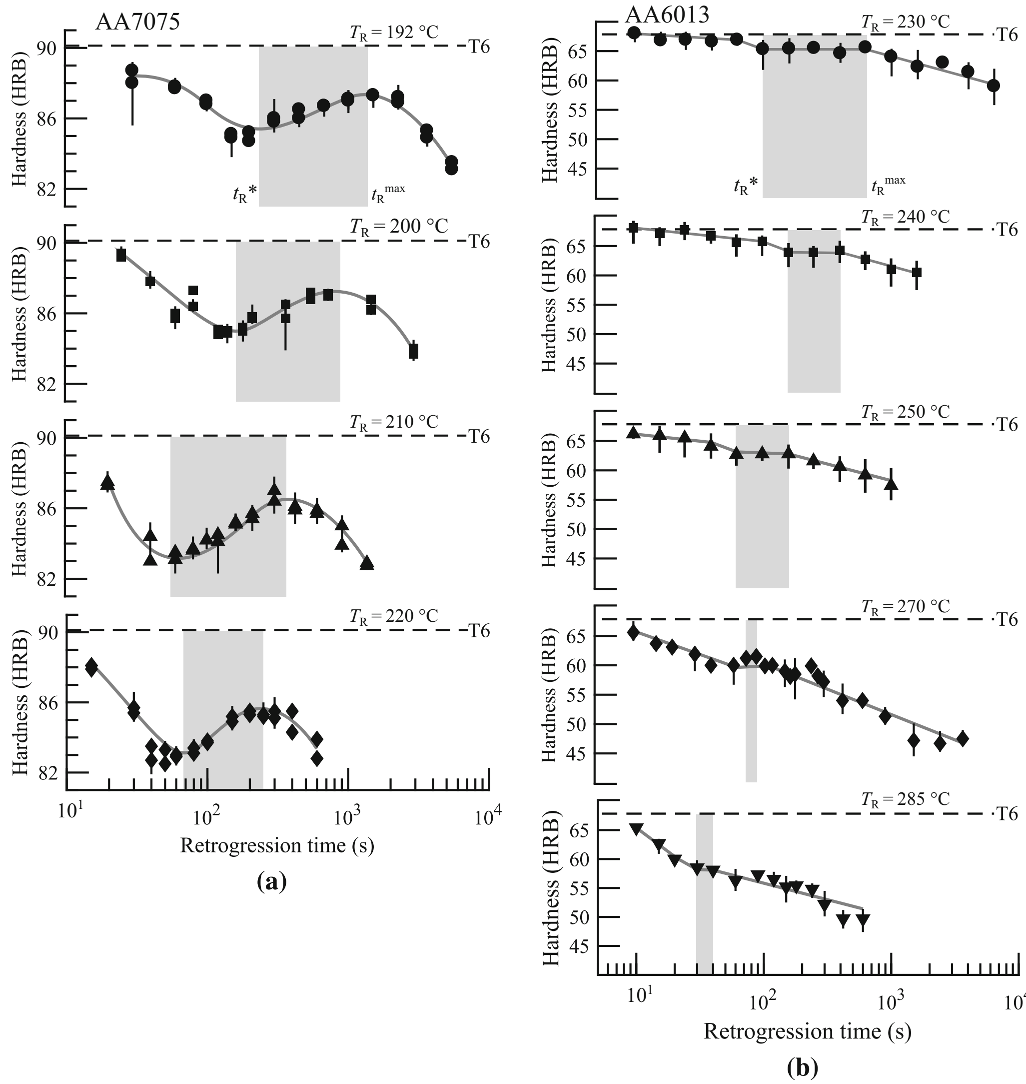

Fig. 3—Data for room-temperature hardness (HRBS) after retrogression are plotted as a function of the logarithm of retrogression time for (*a*) AA7075 and (*b*) AA6013 for several retrogression temperatures, T_R. Symbols show mean values, and error bars designate maximum and minimum values within the measurement sample.

Table VI. Measured Critical Retrogression Times, t_R* and t_R^{max}, at Various Retrogression Temperatures, T_R

Alloy	T_R (°C)	t_R* (s)	t_R^{max} (s)
AA7075	192	237	1358
	200	164	858
	210	56	363
	220	68	247
AA6013	230	100	630
	240	160	400
	250	63	160
	270	75	90
	285	30	40

temperature are listed in Table VI. In general, t_R* and t_R^{max} decrease as retrogression temperature, T_R, increases.

C. *Reaging Heat Treatments*

Figure 4(a) presents the room-temperature hardness of retrogressed-and-reaged AA7075 (AA7075-RRA) specimens. The region from t_R* to t_R^{max} is shaded in gray. The reaging heat treatment used for AA7075 is 120 °C for 24 hours.[5,7] The mean hardness of AA7075-RRA specimens retrogressed for a time less than t_R^{max} is 90.4 HRBS, which is within the measurement uncertainty for the mean hardness of the original T6 temper condition, 90.1 ± 0.4 HRBS. For specimens retrogressed longer than t_R^{max}, the reaging heat treatment does not fully recover all the hardness lost during retrogression.

Figure 4(b) presents the room-temperature hardness of retrogressed-and-paint-baked AA7075 (AA7075-RPB) specimens. The region from t_R* to t_R^{max} is shaded in gray. A reaging heat treatment of 185 °C for 25 minutes was used to simulate the paint-bake process.[7,13,30,31] Across all retrogression times and temperatures studied, reaging with the simulated paint-bake produced a lower mean hardness than reaging with the recommended reaging heat treatment. These differences are larger than the measurement uncertainty of each mean hardness. The mean hardness of AA7075-RPB specimens retrogressed for a time less than t_R^{max} is 88.0 HRBS, which is 2 pct less than the mean hardness of the original T6 temper condition, 90.1 ± 0.4 HRBS. For AA7075, the simulated paint-bake heat treatment is less effective than the reaging heat treatment conducted at 120 °C. For specimens retrogressed longer than t_R^{max}, the RPB hardness further decreases as retrogression time increases.

Figure 4(c) presents the room-temperature hardness of retrogressed-and-paint-baked AA6013 (AA6013-RPB) specimens. The region from t_R* to t_R^{max} is shaded in gray. The mean hardness of AA6013-RPB specimens retrogressed for a time less than t_R* is 68.8 HRBS, which is within the measurement uncertainty for the mean hardness of the original T6 temper condition, 67.8 ± 1.4 HRBS. The mean hardness of AA6013-RPB specimens retrogressed for times ranging from t_R* through t_R^{max} is 67.2 HRBS, which is within the measurement uncertainty for the mean hardness of the original T6 temper condition. These data confirm that AA6013 exhibits retrogression and reaging behavior and suggest that the simulated paint-bake is an effective reaging heat treatment for AA6013. For AA6013-RPB specimens retrogressed longer than t_R^{max}, hardness decreases as retrogression time increases. Hardness is not fully recovered in specimens of AA6013 retrogressed for times longer than $t_R{}^{max}$.

Five different reaging heat treatments were studied for AA6013, listed in Table IV, including the simulated paint-bake heat treatment 185 °C for 25 minutes. The mean final hardness data after retrogression at 240 °C for 400 seconds and reaging by one of these five heat treatments are presented in Figure 5. The mean hardness immediately after retrogression at 240 °C for 400 seconds, labeled as R in Figure 5, is 64.7 HRBS, which is 5 pct less than the mean hardness of the original T6 temper condition, 67.8 ± 1.4 HRBS. The mean hardness after reaging by the simulated paint-bake heat treatment 185 °C for 25 minutes, labeled as RPB in Figure 5, is 68.4 HRBS, which is within the measurement uncertainty for the mean hardness of the original T6 temper condition. The mean hardness after reaging at 190 °C for 1 hour (60 minutes) is 68.8 HRBS, which is 1 pct greater than the mean hardness of the original T6 temper condition and is slightly greater than the hardness of the RPB condition. However, these differences are less than the respective measurement uncertainties for the T6 and RPB conditions. Reaging at 190 °C for 2 to 4 hours produced a mean hardness of 67.3 HRBS. This is within the measurement uncertainty for the mean hardness of the original T6 temper condition but is less than the mean hardness after reaging for only 1 hour. Of the reaging heat treatments

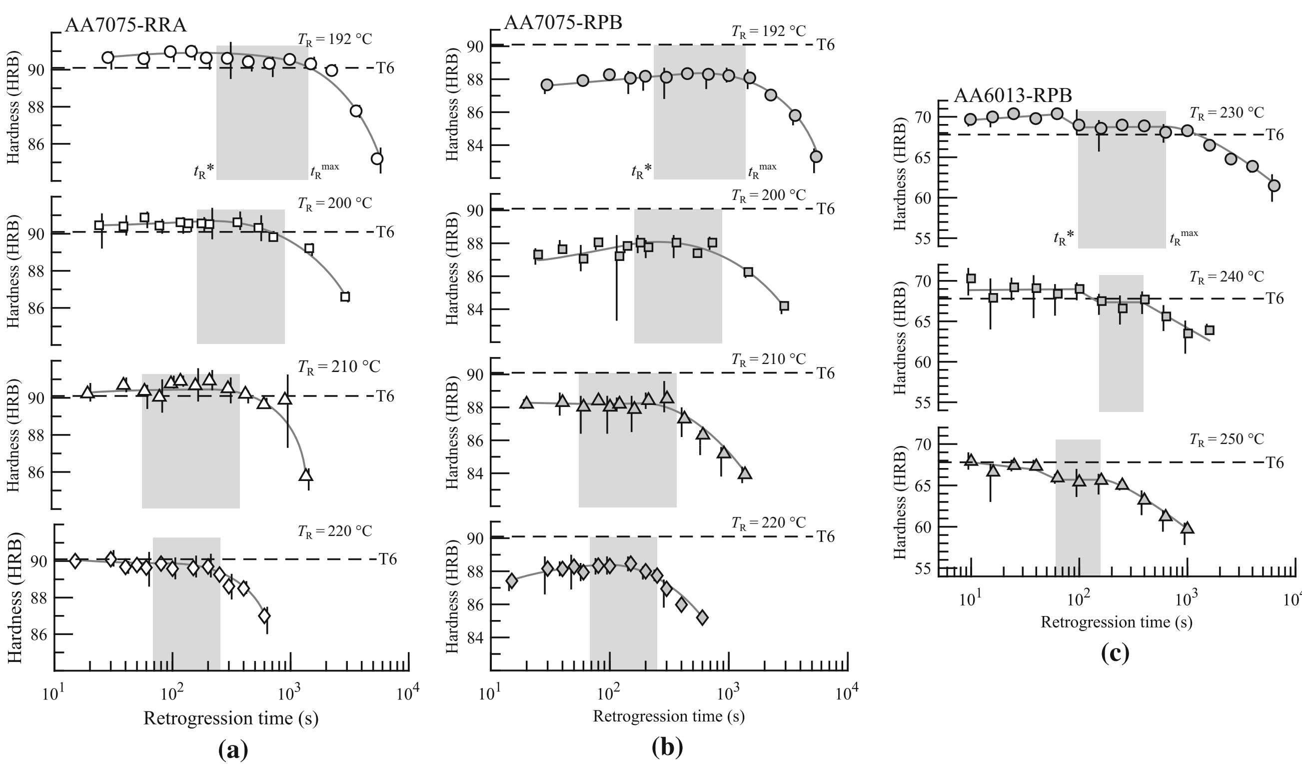

Fig. 4—Data for room-temperature hardness (HRBS) are plotted against the logarithm of retrogression time for (*a*) specimens of AA7075 retrogressed and then reaged at 120 °C for 24 h, (*b*) specimens of AA7075 retrogressed and then reaged with a simulated paint-bake of 185 °C for 25 min, and (*c*) specimens of AA6013 retrogressed and then reaged with a simulated paint-bake of 185 °C for 25 min. Symbols show mean values, and error bars designate maximum and minimum values within the measurement sample.

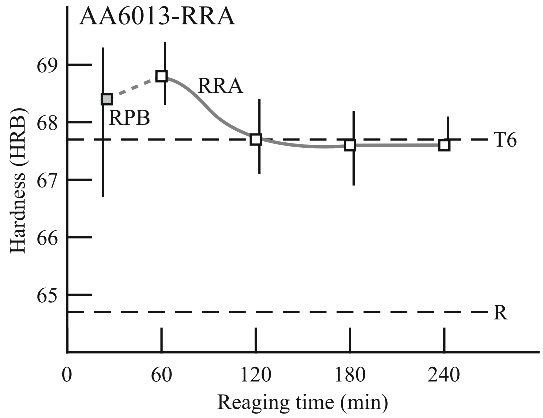

Fig. 5—Room-temperature hardness data (HRBS) of AA6013 after retrogression and reaging are plotted against reaging time. The material was retrogressed from the T6 condition at 240 °C for 400 s and then reaged at either 185 °C for 25 min (RPB) or 190 °C for 60 to 240 min. The recommended reaging heat treatment, 190 °C for 60 min, is labeled RRA. Symbols show mean values, and error bars designate maximum and minimum values within the measurement sample.

studied, 190 °C for 1 hour is recommended as a reaging heat treatment for AA6013 and is labeled as RRA in Figure 5.

The retrogressed (R), retrogressed-and-paint-baked (RPB), and retrogressed-and-reaged (RRA) room-temperature hardness data of AA7075 are plotted as functions of retrogression time in Figure 6(a). The region from t_R* to t_R^{max} is shaded in gray. All the specimens were retrogressed at 200 °C. Specimens in the RPB condition were reaged with the simulated paint-bake heat treatment of 185 °C for 25 minutes.[7,13,30,31] Specimens in the RRA condition were reaged at 120 °C for 24 hours.[5,7] The mean hardness of RPB specimens retrogressed for a time shorter than t_R^{max}, 858 seconds at 200 °C, is 87.7 HRBS. This is 3 pct less than the mean hardness of the original T6 temper condition, 90.1 ± 0.4 HRBS. The mean hardness of RRA specimens retrogressed for times shorter than t_R^{max} is 90.5 HRBS. This is within the measurement uncertainty for the mean hardness of the original T6 temper condition, 90.1 ± 0.4 HRBS, and is greater than specimens reaged with a simulated paint-bake heat treatment. For specimens retrogressed longer than t_R^{max}, the simulated paint-bake heat treatment produces no recovery of hardness greater than the measurement uncertainty. For specimens retrogressed longer than $t_R{}^{max}$, the full reaging heat treatment increases hardness by 2.8 HRBS, on average. However, the RRA hardness of these specimens is always less than the mean hardness of the original T6 temper condition.

The retrogressed (R), retrogressed-and-paint-baked (RPB), and retrogressed-and-reaged (RRA) room-temperature hardness data for AA6013 are plotted as functions of retrogression time in Figure 6(b). The region from t_R* to t_R^{max} is shaded in gray. All of the specimens were retrogressed at 240 °C. Specimens in the RPB condition were reaged with the simulated paint-bake heat treatment 185 °C for 25 minutes.[7,13,30,31] Specimens in the RRA condition were reaged at 190 °C for 1 hour, the recommended reaging heat treatment established in Figure 5. The mean hardness of RPB specimens retrogressed for times shorter than t_R*, 160 seconds at 240 °C, is 69.0 HRBS, which is 2 pct greater than the mean hardness of the original T6 temper condition, 67.8 ± 1.4 HRBS. However, this difference is less than the measurement uncertainty for the mean hardness of the original T6 temper condition. The mean RRA hardness of the specimen retrogressed at 240 °C for 100 seconds, which is shorter than t_R*, is also 69.0 HRBS. These data demonstrate that for retrogression times shorter than t_R*, both the paint-bake and the recommended reaging heat treatment are effective reaging heat treatments for AA6013. The mean hardness of RPB specimens retrogressed for times from t_R* through t_R^{max}, 160 through 400 seconds at 240 °C, is 67.3 HRBS, which is within the measurement uncertainty for the mean hardness of the original T6 temper condition. The mean RRA hardness of the specimen retrogressed at 240 °C for t_R^{max}, 400 seconds, is 68.8 HRBS. While this mean hardness is greater than both the mean hardness of the original T6 temper condition and the mean RPB hardness of the specimen retrogressed at 240 °C for 400 seconds, these differences are within the measurement uncertainties for both the T6 and RPB conditions. These data demonstrate that for retrogression times from t_R* through t_R^{max}, the recommended reaging heat treatment for AA6013 may be more effective than the simulated paint-bake heat treatment. For RPB specimens retrogressed longer than t_R^{max}, hardness decreases as retrogression time increases. For a retrogression time of 1000 seconds, slightly longer than t_R^{max}, the recommended reaging heat treatment produces a greater hardness than reaging with the simulated paint-bake, and this difference in hardness is larger than the measurement uncertainty. However, even the recommended reaging heat treatment barely recovers the T6 hardness. Retrogression times longer than t_R^{max} should be avoided to prevent an unrecoverable loss of hardness.

Throughout the retrogression times investigated, the mean hardness data of AA6013-RPB specimens are greater than the corresponding retrogressed specimens, even at very short retrogression times, see Figure 6(b). These data demonstrate that the simulated paint-bake heat treatment is consistently beneficial to retrogressed specimens of AA6013. This effect does not occur for AA7075, see Figure 6(a). At very short retrogression times, the mean hardness data of AA7075-RPB specimens are less than the retrogressed condition. At very long retrogression times, the simulated paint-bake heat treatment does not substantially increase hardness. These data demonstrate that for very short and for very long retrogression times, the simulated paint-bake heat treatment is not beneficial to the hardness of retrogressed specimens of AA7075.

IV. DISCUSSION

Ivanoff *et al.* hypothesized that retrogression is a thermally activated process that can be characterized

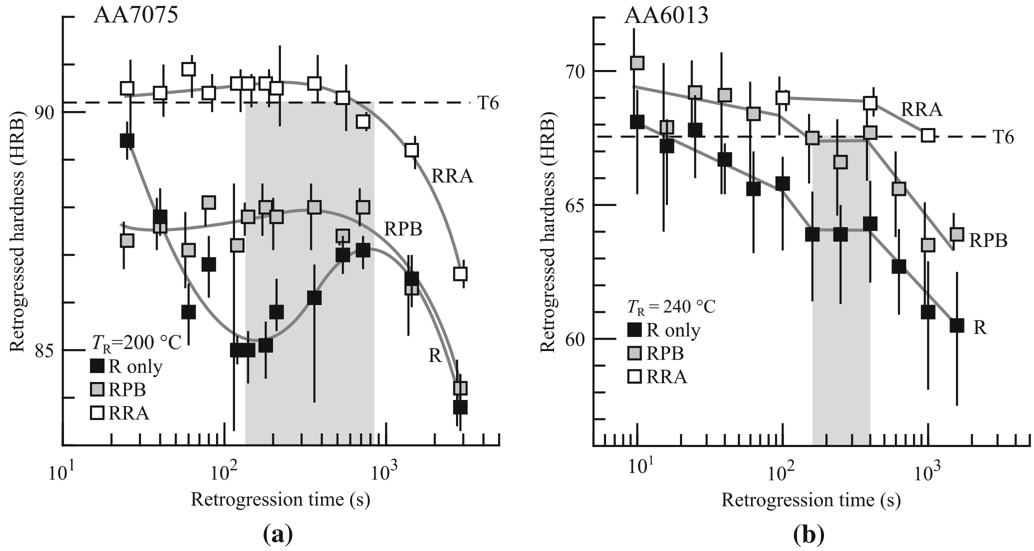

Fig. 6—Room-temperature hardness data (HRBS) are plotted for (*a*) AA7075 and (*b*) AA6013 against the logarithm of retrogression time. Specimens of AA7075-T6 were retrogressed at 200 °C and then reaged with either a simulated paint-bake heat treatment (185 °C for 25 min) or the recommended reaging heat treatment (120 °C for 24 h). Specimens of AA6013-T6 were retrogressed at 240 °C and then reaged with either a simulated paint-bake heat treatment (185 °C for 25 min) or the recommended reaging heat treatment (190 °C for 1 h). Symbols show mean values, and error bars designate maximum and minimum values within the measurement sample.

with a single activation energy when the diffusion of a single species controls the rate of retrogression.[7] Although Park and Ardell identified multiple precipitate reactions that occur during the retrogression of AA7075,[8] Ivanoff *et al*. successfully described retrogression in AA7075 using a single activation energy of 95 kJ/mol.[7] This is the activation energy for precipitate dissolution in Al-Mg-Zn alloys measured by Baldarach *et al*.[31] Ivanoff *et al*. concluded that the diffusion of a single species, thought to be Zn, controls the complex precipitate reactions observed by Park and Ardell.[8] Ivanoff *et al*. calculated reduced retrogression time, see Eq. [1], to accurately describe the change in room-temperature hardness of AA7075 throughout retrogression for different times and temperatures. In the present study, activation energies for retrogression are directly measured for AA7075 and AA6013 using DSC. For each material, all of the specimens used to measure the activation energy of retrogression were taken from the same manufacturing lot and were heat treated together. Table V reports the mean endothermic peak temperature measured from DSC data for each material and heating rate investigated. Using the following relationship developed by Barczy and Tranta, the activation energy for retrogression is calculated,[49]

$$\ln\left(\frac{T_{\mathrm{P}} - T_0}{\beta}\right) - \frac{Q_{\mathrm{R}}}{R}\frac{1}{T_{\mathrm{P}}} = B, \qquad [3]$$

where T_{P} is the temperature at which the endothermic peak occurs, T_0 is the starting temperature for the DSC experiments (25 °C), β is the heating rate, Q_{R} is the activation energy for retrogression, R is the universal gas constant, and B is a material constant.[49] When the natural logarithm of $\left(\frac{T_P - T_0}{\beta}\right)$ is plotted as a function of the inverse peak temperature, T_{P}^{-1}, the slope of the data is equal to Q_{R}/R, as demonstrated in Figure 7.

From the data presented in Figure 7(a), the activation energy of retrogression for AA7075-T6 is measured as 97 ± 7 kJ/mol. This activation energy is consistent with the activation energy for the dissolution of precipitates in an Al-Mg-Zn alloy.[31] From the data presented in Figure 7(b), the activation energy of retrogression for AA6013-T6 is measured as 160 ± 30 kJ/mol. The uncertainty of each activation energy is estimated from the 95 pct confidence interval of the slope fitted to the data in Figure 7.[47]

Previous investigations by Starink[43] and by Braun[44] determined that the endothermic peaks identified in Figure 2 and Table V correspond with the dissolution of each alloy's respective strengthening precipitates. For AA7075, the endothermic peaks identified correspond with the dissolution of η' precipitates.[43] This endothermic peak is followed by an exothermic valley that corresponds with the precipitation of η precipitates.[43] Park and Ardell determined that for AA7075, one of the first reactions to occur during retrogression is the dissolution of η' precipitates, which correlates with the initial loss of strength demonstrated in Figure 1.[8] For AA7075-T651 the η' precipitates are plate shaped and are on the order of 5 to 6 nm in diameter.[50] The η precipitates also tend to be plate shaped and are on the order of 4 to 10 nm in diameter.[50] For AA6013, the endothermic peaks identified in Figure 2 and Table V correspond with the dissolution of β'' and Q'

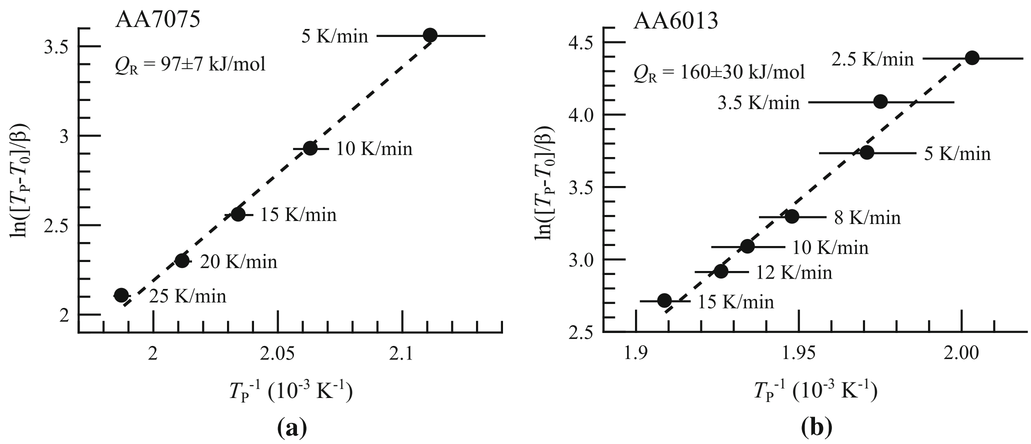

Fig. 7—The activation energies of retrogression in (*a*) AA7075-T6 and (*b*) AA6013-T6 are measured from the slope of DSC data plotted as the natural logarithm of $(T_P - T_0/\beta)$ *vs* the inverse peak temperature, T_P^{-1}.

precipitates.[44] These precipitates are needle and lathe shaped, respectively, and are on the order of 6 to 10 nm in length in AA6013-T6.[44–46]

The critical retrogression times measured from hardness data, see Table VI, generally decrease as temperature increases. Ivanoff *et al.* proposed the concept of reduced retrogression time to account for both retrogression time and retrogression temperature using the Arrhenius relationship defined in Eq. [1].[7,34] Plotting retrogressed hardness against reduced retrogression time aligns the values of t_R* at different temperatures to a single reduced retrogression time of τ_R* for all temperatures. Likewise, the different values of t_R^{max} at different temperatures are aligned to a single reduced retrogression time of τ_R^{max}. These are demonstrated in Figure 8. For AA7075, the critical reduced retrogression times are $\tau_R* = (2.9 \pm 0.9) \times 10^{-9}$ seconds and $\tau_R^{max} = (1.5 \pm 0.3) \times 10^{-8}$ seconds. For AA6013, the critical reduced retrogression times are $\tau_R* = (1.6 \pm 1.5) \times 10^{-14}$ seconds and $\tau_R^{max} = (2.6 \pm 1.4) \times 10^{-14}$ seconds. These critical reduced retrogression times are the means of the critical retrogression times listed in Table VI converted into reduced retrogression time. The uncertainty of each critical reduced retrogression time is presented as the total range in data divided by two. The regions from τ_R* to τ_R^{max} are shaded as gray in Figure 8. For AA6013, the span in the logarithm of reduced retrogression time from τ_R* to τ_R^{max} is very short. Additionally, the measurement uncertainties of the critical reduced retrogression times for AA6013 are quite large. For practical purposes, the plateau region between the critical reduced retrogression times for AA6013 is considered to be an inflection point on the master curve. Retrogression for times beyond this inflection point produce an unrecoverable loss of hardness.

The critical reduced retrogression times for AA7075 and AA6013 are used to predict critical retrogression times for a range of retrogression temperatures in Figure 9. The predicted critical retrogression times are indicated by dashed lines. The region from predicted t_R* to predicted t_R^{max} is shaded in gray. The measured critical retrogression times are plotted as individual data points. For AA7075, see Figure 9(a), the dashed lines of predicted critical retrogression times closely match the experimental data. For AA6013, see Figure 9(b), the dashed line for predicted t_R^{max} matches the experimental data reasonably well, but the dashed line for predicted t_R* does not. Regardless, the span from t_R* to t_R^{max} is quite narrow. This reinforces the interpretation of the short plateau in AA6013 hardness, see Figure 8(b), as an inflection point on the master curve. So long as t_R is less than t_R^{max}, a single reaging heat treatment can recover the hardness lost during retrogression of AA6013.

Recommendations for retrogression heat treatments are made from the predicted critical retrogression times in Figure 9. The recommended retrogression heat treatment for AA7075-T6 is 200 °C for times from 3 to 12 minutes. The recommended reaging heat treatment for AA7075 is 120 °C for 24 hours, as previously documented by other investigators.[5,7] After the recommended retrogression heat treatment, reaging by the simulated paint-bake heat treatment increases room-temperature hardness but is not as effective as the recommended reaging heat treatment. For AA6013, the narrow range between the two critical reduced retrogression times is interpreted as an inflection point on the master curve of Figure 8(b). The recommended retrogression heat treatment for AA6013-T6 is 240 °C for 7 minutes. The recommended reaging heat treatment for AA6013 is 190 °C for 1 hour. This reaging heat treatment provides the greatest room-temperature hardness among the reaging heat treatments studied. Reaging using the simulated paint-bake heat treatment is only slightly less effective than this recommended reaging heat treatment.

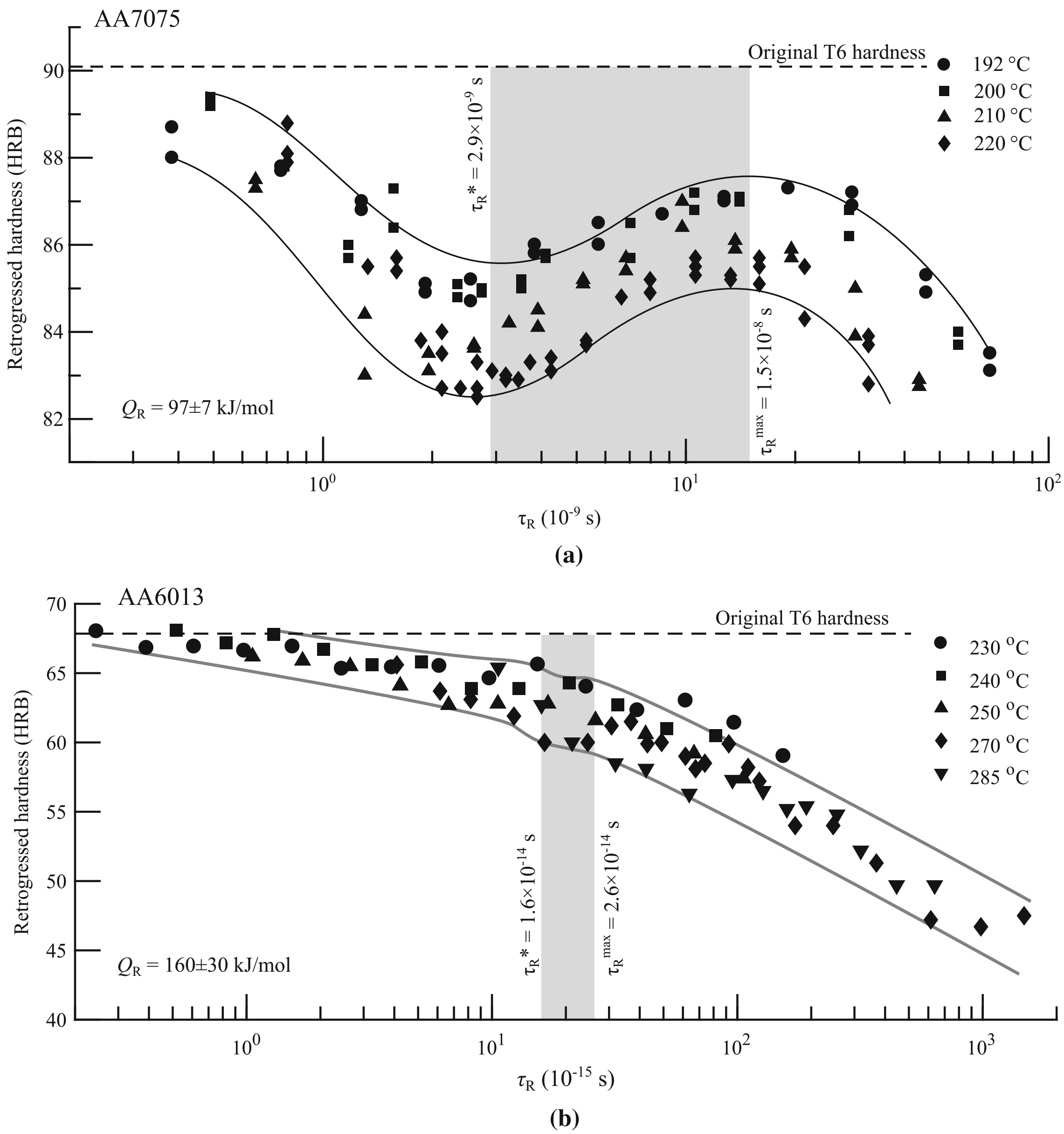

Fig. 8—Master curves are produced by plotting room-temperature hardness after retrogression as a function of reduced time. (*a*) is the master curve for AA7075-T6, where reduced time is calculated using the activation energy Q_R = 97 kJ/mol. (*b*) is the master curve for AA6013-T6, where reduced time is calculated using the activation energy Q_R = 160 kJ/mol. For clarity, error bars are not shown.

V. CONCLUSIONS

1. The activation energies for retrogression are 97 ± 7 kJ/mol for AA7075-T6 and 160 ± 30 kJ/mol for AA6013-T6, as measured using differential scanning calorimetry.
2. The recommended retrogression heat treatment for AA7075-T6 is 200 °C for 3 to 12 minutes.
3. Reaging at 120 °C for 24 hours after the recommended retrogression heat treatment restores the hardness of the original T6 temper condition for AA7075.
4. Reaging at 185 °C for 25 minutes, which simulates a paint-bake heat treatment, after the recommended retrogression heat treatment produces a hardness that is 3 pct less than the mean hardness of the original T6 temper condition for AA7075.
5. AA6013-T6 demonstrates a significant retrogression and reaging response. RRA behavior has not been previously reported for AA6013-T6 by other investigators.
6. Rather than producing a local minimum and a local maximum in hardness after retrogression, AA6013-T6 produces a short plateau. The beginning and end times of this plateau are defined as t_R* and t_R^{max}, respectively.

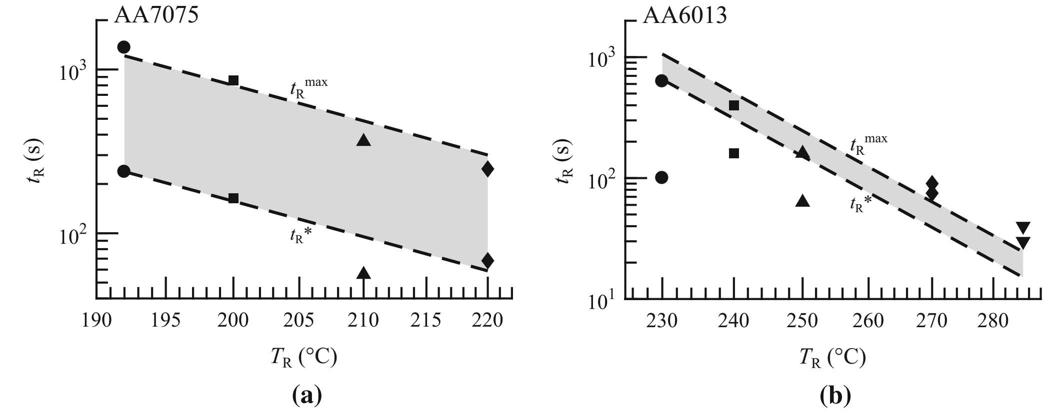

Fig. 9—Predicted retrogression time is plotted as a function of retrogression temperature for (*a*) AA7075 and (*b*) AA6013 on dual-logarithmic scales. The shaded region ranges from predicted t_R* to predicted t_R^{max}. Experimentally measured values of t_R* and t_R^{max} are plotted as individual data points.

7. The recommended retrogression heat treatment for AA6013-T6 is 240 °C for 7 minutes.
8. The recommended reaging heat treatment for AA6013-T6 following the recommended retrogression heat treatment is 190 °C for 1 hour. This reaging heat treatment produces a hardness that is 1 pct greater than and within the measurement uncertainty for the mean hardness of the original T6 temper condition.
9. Reaging at 185 °C for 25 minutes, which simulates a paint-bake heat treatment, after the recommended retrogression heat treatment produces a final hardness that is 1 pct greater than the mean hardness of the original T6 temper condition for AA6013. This simulated paint-bake heat treatment is nearly as effective as the recommended reaging heat treatment for AA6013.
10. Experimental data are used to calculate critical reduced retrogression times, τ_R* and τ_R^{max}. The critical reduced retrogression times of AA7075 are $\tau_R^* = (2.9 \pm 0.9) \times 10^{-9}$ seconds and $\tau_R^{max} = (1.5 \pm 0.3) \times 10^{-8}$ seconds. The critical reduced retrogression times of AA6013 are $\tau_R^* = (1.6 \pm 1.5) \times 10^{-14}$ seconds and $\tau_R^{max} = (2.6 \pm 1.4) \times 10^{-14}$ seconds. For AA6013, the span from τ_R* through τ_R^{max} is interpreted as an inflection point.
11. Reduced retrogression time is successfully used to design retrogression heat treatments for AA7075-T6 and AA6013-T6 using the graphical approach presented in Figure 9.

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