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The effect of cooling rate on the quench sensitivity of 2618 Al/Al_2O_3 MMC

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Metal matrix composites (MMCs) are materials consisting of metal alloys reinforced with fibers, whiskers, particulates, and wires. Due to their superior mechanical properties, such as low coefficients of thermal expansion and high specific stiffness, they are attractive for many structural and non-structural applications. The most notable production applications are found in the aerospace, automobile, and sports equipment industries. Despite the great potentials possessed by MMCs, there are some concerns regarding the effect of the reinforcements, which are mostly ceramics, on the properties of the matrix alloys. One such property is the quench sensitivity of the matrix material. Heat treatable aluminum alloys are quench sensitive (i. e. their properties and precipitation behavior change with cooling rate or quenchant). The rate of cooling or the type of quenchant used during the fabrication process or the subsequent solution heat treatment affects the mechanical properties of these materials. Therefore, any modification that can alter the quench sensitivity significantly could have important consequences on the heat treatment of the alloys. Thus, the quench parameters may have to be more tightly controlled than for the unreinforced alloy in order to maintain consistent as-quenched properties. In the present study, the quench sensitivity of 2618 Al alloy and its composite containing 10 vol. % Al_2O_3 particles was investigated using hardness measurements and differential scanning calorimetry (DSC). Although 2618 Al is quench sensitive, its quench sensitivity was significantly increased by the addition of Al_2O_3 particles. Also, cooling rate affected the precipitation kinetics and the volume fraction of the precipitate phases formed in both materials. © *2000 Kluwer Academic Publishers*

1. Introduction

Within the last decade, metal matrix composites (MMCs) have emerged as viable materials for a variety of applications that span a wide spectrum of industries (e.g., aerospace, electronic packaging, automobile, and sports equipment). MMCs, depending on the material system and applications, show significant improvements in mechanical properties above those that would be obtained by conventional alloying techniques. These include reduced structural weight, improved stiffness, improved dynamic response, reduced wear, and reduced coefficients of thermal expansion. A great deal of research effort has been devoted to study the mechanical properties and the microstructures of these materials during aging [1–8]. However, investigations of their quench sensitivity have received much less attention [9–11].

A limiting factor in producing heavy sections of high strength aluminum alloys is their quench sensitivity, which implies a change in the as-quenched properties and precipitation behavior with cooling rate. Many aluminum alloys [9–18] and aluminum-based MMCs [9–11] are known to be quench sensitive, although the sensitivity differs between alloy systems. The cooling rate or the type of quenchant used during quenching from the solution heat treatment temperature affects the mechanical properties of age-hardenable aluminum alloys by altering the precipitation behavior of the alloy.

Quench sensitivity is particularly of great concern in aluminum alloys in which the amounts of alloying elements are very close to the solubility limit at the solution heat treatment temperature. The response to a particular quench rate is controllable. In particular, it has been observed that the presence of minor addition elements (MAEs) such as chromium, manganese, vanadium, and zirconium (which are used in commercial alloys to control grain size and recrystallization or increase strength) promote quench sensitivity [14, 15]. Thompson *et al.* [14] found that whereas very low levels of MAEs can cause considerable quench sensitivity, high levels of

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soluble alloying elements alone do not produce quench sensitivity. According to them, alloys containing very low levels of MAEs produce fine intermetallic particles which act as nucleation sites for large precipitates during slow cooling. Amongst the aluminum alloys containing MAEs that they studied, manganese- and zirconium-containing alloys produced the least sensitivity while those bearing chromium and vanadium suffered up to 50% reduction in strength. They proposed that an embrittlement failure mechanism found in alloys containing manganese or very low levels of MAEs was responsible for the magnitude of quench sensitivity measured in such materials. On the other hand, Conserva and Fiorini [13], who used transmission electron microscopy (TEM) and electrical resistivity measurements to study the effect of chromium and zirconium on the quench sensitivity of Al-Zn-Mg-Cu alloys, reported that the presence of chromium and zirconium did not affect the aging process of the alloy after fast quenching. Also, there was no decrease in Guinier-Preston (GP) zone density due to their presence. Rather, they found that the aging response of the alloys studied strongly depended on the precipitation induced by quenching (the cooling rate differences only determined a variation of super-saturation of the alloys and of the vacancy concentration). Also, they observed that for the same quenching conditions, there is a relationship between the density and size of quench-induced precipitates and the presence of alloying elements. Chromium-containing alloys form denser and coarser $MgZn_2(\eta)$ particles. Hence, they rejected the notion that other factors are required to account for the quench sensitivity phenomenon except the solute fraction precipitated during the quenching process (measured in terms of GP zone density).

Investigations using differential scanning calorimetry (DSC) [9] and hardness measurements [10] have been carried out to study the quench sensitivity phenomenon in discontinuously reinforced MMCs. Both studies showed that the MMCs are more quench sensitive than the unreinforced alloys. In fact, normally quench insensitive materials such as 6061 aluminum became quench sensitive due to the presence of ceramic reinforcements [9]. In addition, by making use of extensive literature data, Thomas and King [10], who studied the PM 2124 alloy and its composites, reported that the unreinforced 2124 alloy exhibited two different quench sensitivity regimes, whereas the MMCs showed only one sensitivity regime. The present work deals with the effect of cooling rate resulting from using different quenchants, on the as-quenched hardness and the subsequent natural and artificial aging behavior of ingot metallurgy (IM) 2618 aluminum alloy and its composite containing 10 vol. % alumina (Al_2O_3) particles.

2. Materials

An ingot metallurgy (IM) 2618 aluminum reinforced with 10 vol. % Al_2O_3 particles was used in this investigation. Duralcan Inc. (San Diego, USA) supplied the material, which is no longer in production, in the form of extruded rectangular bars. The unreinforced 2618 aluminum, processed by the same route and also supplied by the same manufacturer, was used as a reference. The nominal chemical composition of both materials is listed in Table I.

3. Experimental procedure

The quench sensitivity of the two materials was monitored by microhardness measurements. Rectangular samples were solution heat-treated at $530 \pm 2\,^{\circ}C$ for two hours, quenched in different media - air, hot water ($100\,^{\circ}C$), room temperature (RT)/laboratory water, cold methanol ($-30\,^{\circ}C$) and liquid nitrogen -, aged naturally for 14 days, and subsequently precipitation-hardened at $200 \pm 1\,^{\circ}C$ for up to 50 h using a constant-temperature air furnace. The Vickers microhardness was measured on polished samples using a Buehler Microhardness Tester - Micromet II - with a load of 100 g applied for 15 seconds. The small load was chosen to produce indentations small enough to occur only in the particle-free matrix. Each hardness value reported was the average of at least six measurements. The presence of any subsurface particles and voids was identified by excessively high or low hardness values, respectively, which were discarded.

The quench sensitivity of both materials was also investigated using the DSC. Small slices were cut from the extrudates from which discs (approximately 5-mm diameter, 1–1.2 mm thick) were prepared. The discs were solution heat-treated and quenched in the same manner as the hardness samples, but they were neither aged naturally nor artificially after quenching. DSC tests were conducted on each material in the as-quenched condition using a Mettler TA 4000 thermal analyzer (TA) equipped with a DSC 30 cell. At least, two samples of each material were used to ensure reproducibility. The DSC scans were initiated at $30\,^{\circ}C$ and completed at $520\,^{\circ}C$. Other details about the experiment have been given elsewhere [19].

4. Results and discussion

Figs 1 and 2 show the variation of as-quenched and T4 (naturally aged) hardness with cooling rate, respectively, for 2618 and $2618 + 10$. Some interesting features can be seen in both figures. In Fig. 1, the as-quenched hardness values of the air-cooled samples are higher than those of the samples supposedly quenched at relatively higher cooling rates in hot water, laboratory water, and cold ethanol. Only samples quenched in

TABLE I Chemical compositions of test materials

	Element (wt. %)									
Material*	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Al_2O_3
2618	0.18	1.19	2.34	----	1.59	----	1.05	----	0.07	----
$2618 + 10$	0.17	1.15	2.15	0.01	1.69	0.002	1.08	0.02	0.07	9.3[†]

*2618 = 2618 Al; and $2618 + 10$ = 10 vol. % Al_2O_3/2618 Al composite. [†]Composition in vol. %. Balance = Al.

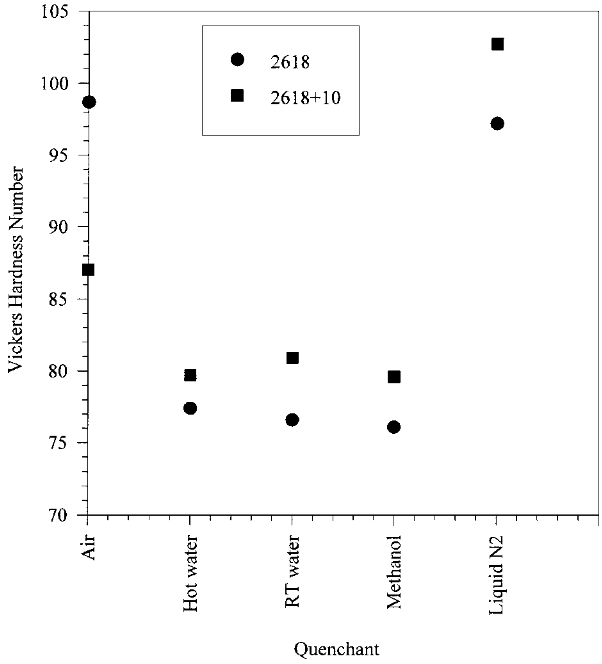

Figure 1 Variation of as-quenched hardness with cooling rate for 2618 and 2618 + 10.

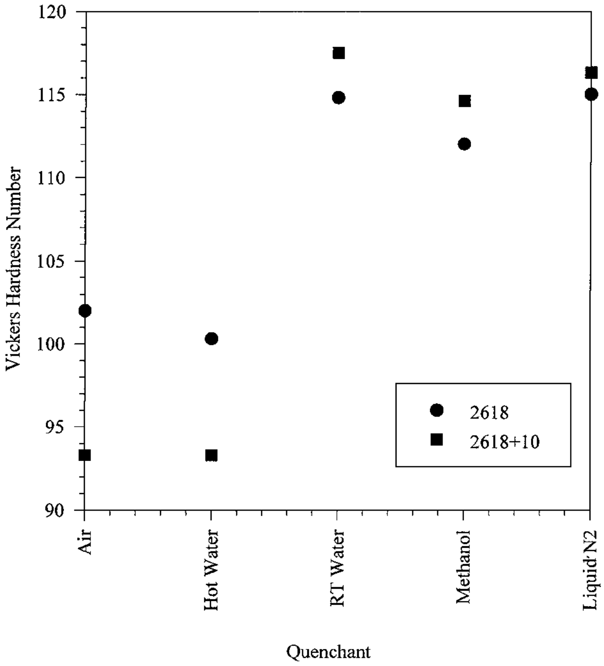

Figure 2 Effect of cooling rate on samples aged naturally for 14 days.

liquid nitrogen exhibited higher hardness. As observed in other studies [9–11], this is an indication that precipitates other than Guinier-Preston-Bagaryatskii (GPB) zones are formed in significant amounts during air cooling. On the other hand, hardening at higher cooling rates is due to vacancy/GPB zones and dislocations. As the cooling rate increases, the amount of quenched-in vacancies that is necessary for GPB zones formation also increases. However, in the composite, the increased cooling rate also increases the dislocation density.

Flom and Arsenault [20] have reported that higher cooling rates induce greater elastic straining in the vicinity of reinforcement particles than in the bulk matrix. Consequently, the density of dislocations generated to relieve this straining is likely to increase proportionately. The dislocation density of MMCs has been modeled in terms of the temperature difference of quenching (ΔT) [21, 22].

Dislocations and matrix-particle interfaces serve as vacancy sinks, which inhibit GPB zone formation [4]. Also, solute depletion at the particle-matrix interface retards GPB zone formation. Nevertheless, Fig. 1 shows that the as-quenched hardness of the MMC samples cooled at high cooling rates (in hot water, room temperature water, cold ethanol, and liquid nitrogen) is higher than that of the unreinforced alloy. This is attributed to the large amount of dislocations formed in the MMC rather than to quenched-in vacancies and GPB zones since the effect of cooling rate on GPB zone formation is smaller in MMCs.

Fig. 2 reveals other features that are not very obvious from Fig. 1. It consists basically of two regimes. After aging naturally for 14 days, the hardness of the air- and hot water-cooled composite samples was lower than that of their unreinforced 2618 counterparts, whereas the reverse was the case for specimens quenched in RT water, cold ethanol, and liquid nitrogen. This is interpreted to mean that the unreinforced alloy has larger amounts of GPB zones and vacancies when cooled slowly than the composite. The intermediate precipitates present, the volume fraction of which is expected to be more in the composite than in the monolithic alloy, are coarse and do not contribute significantly to strengthening during aging [13, 16]. Therefore, at low cooling rates, quench sensitivity arises primarily from the decreased GPB zone nucleation capability during subsequent aging due to the reduction of quenched-in vacancies as cooling rate is decreased. At high cooling rates, the amount of quenched-in vacancies available for precipitation and solute strengthening is increased in both the unreinforced alloy and the composite. This, coupled with dislocation strengthening, accounts for the sharp increase in the T4 hardness observed in samples subjected to higher cooling rates. However, a close examination of Fig. 2 shows that samples quenched in laboratory water have slightly higher hardness than those quenched in cold ethanol and liquid nitrogen. This is attributed to the reduction in the concentration of quenched-in vacancies (available for GPB zone formation) as the cooling rate exceeds a certain critical rate after which the probability of vacancy annihilation at dislocations and thermal shock-induced microcracks increases with ΔT.

Figs 3 and 4 show the effect of cooling rate on the aging response at 200 °C for the unreinforced alloy and the composite, respectively. The aging curves show similar trends to those reported in reference [10]. There is no clear indication that the time to peak hardness is affected by quench rate. It can be seen that at high cooling rates, the composite is less quench sensitive than the unreinforced alloy, whereas at low cooling rates, where intermediate precipitate formation dominates, the reverse is the case. It has been reported that decreasing the quench rate reduces the hardness achieved during subsequent aging [13, 16, 23]. This is due to (i) a reduction in the quenched-in vacancies and dislocations which, in turn, causes intermediate precipitates to form

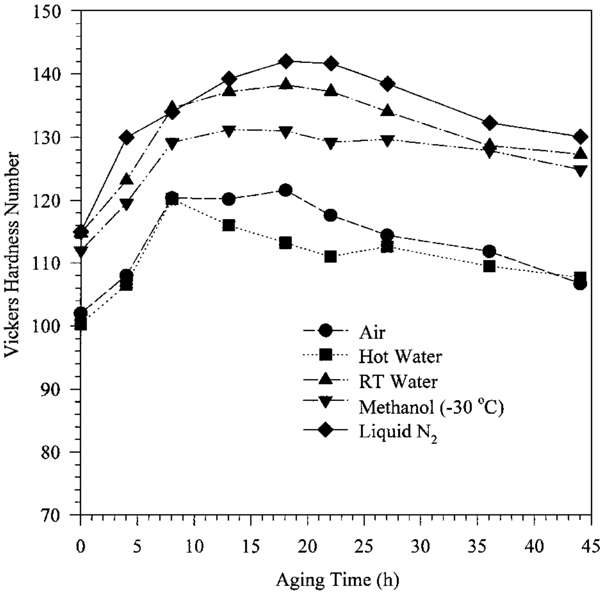

Figure 3 Variation of microhardness with aging temperature and cooling rate (2618).

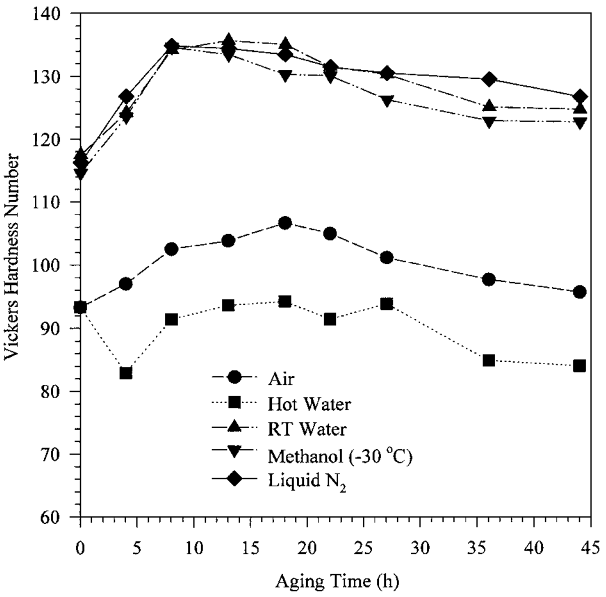

Figure 4 Variation of microhardness with aging temperature and cooling rate (2618 + 10).

on an increasingly coarse scale [23] and (ii) the formation of intermediate precipitates on high temperature intermetallic and, in the present case, reinforcement (Al_2O_3) particles during quenching (especially at low cooling rates), thereby causing less solute to be available for precipitate formation during subsequent aging [13, 16].

Figs 5 and 6 show the DSC thermograms of 2618 and 2618 + 10 samples, respectively, quenched in different media. The first exothermic peak (A) is due to GPB zone formation; the second exothermic doublet peak (C) is due to intermediate phase (S′ and θ') formation; and the endothermic peak (B) represents GPB zone dissolution [6, 8, 9, 19, 24]. It is evident from the figures that the aging sequence of the parent alloy is not affected by either the presence of Al_2O_3 particles or the cooling rate, although the volume fractions of the precipitate phases are significantly altered. The area of

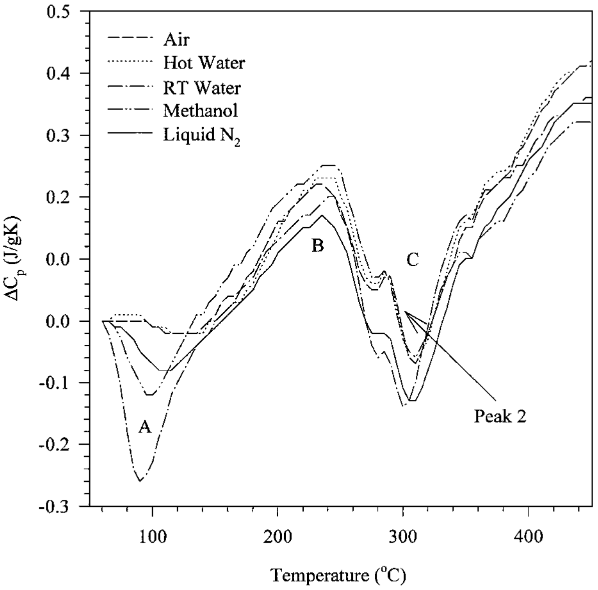

Figure 5 Effect of cooling rate on precipitation reactions in 2618.

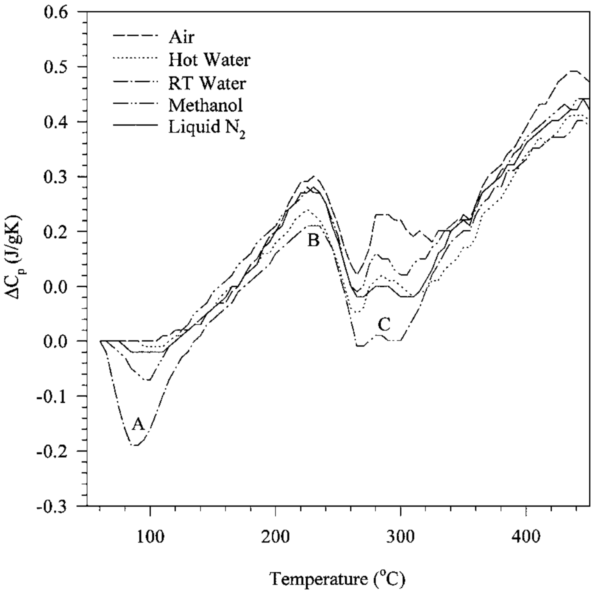

Figure 6 Effect of cooling rate on precipitation reactions in 2618 + 10.

the reaction peak gives the reaction enthalpy, which is directly related to the molar heat of reaction and the volume fraction of the forming or dissolving phase [25], while the temperature is related to the size and stability of the precipitate and the reaction kinetics [26].

It can be seen from Figs 5 and 6 and Tables II and III that the peak reaction temperature and the volume fraction of GPB zones were affected by cooling rate. For

TABLE II Effect of cooling rate on reaction enthalpy

	GPB Zone Formation (J/g)		GPB Zone Dissolution (J/g)	
Quenchant	2618	2618 + 10	2618	2618 + 10
Air	1.2	0	5.4	5.3
Hot Water	1.5	0.96	7.2	5.4
RT Water	6.3	5.2	7.9	6.7
Ethanol	3.1	2.5	6.5	4.3
Liquid N_2	2.4	1.7	6.5	4.8

TABLE III Effect of cooling rate on peak reaction temperature

	GPB Zone Formation (°C)		GPB Zone Dissolution (°C)		Intermediate Phase Formation (Peak 2) (°C)	
Quenchant	2618	2618 + 10	2618	2618 + 10	2618	2618 + 10
Air	125.5*	-	238.4	227.9	309.3	-
Hot Water	109.3*	105.0	237.0	230.0	308.0	310.9
RT Water	90.3	91.7	241.9	233.1	301.5	295.6
Ethanol	99.8	94.1	239.0	229.4	308.7	308.5
Liquid N_2	104.1	102.9	236.2	229.9	308.1	308.3

*Difficult to measure and may contain errors.

example, after air cooling, no GPB zone formation occurred during the DSC scan of the composite material, but the unreinforced alloy showed some presence of GPB zones (about 20% of the GPB zones formed in water-quenched samples). Room temperature water quenching resulted in the formation of more GPB zones than quenching in either cold ethanol or liquid nitrogen. This indicates that at room temperature and below, the heat capacity of the quenchant rather than the temperature or ΔT is the main determining factor in quenching operations. In general, the volume fraction of GPB zone precipitated during DSC scan decreased with decreasing cooling rate for both materials. During slow cooling, there is enough time for portions of the solute to be precipitated at high temperatures. As such, there are less solute atoms in solid solution to contribute to age hardening. This is very evident from the age hardening results shown in Figs 3 and 4. On the other hand, the reaction peak temperature for GPB zone formation tends to increase with decreasing cooling rate. As shown, the reaction peak temperatures shifted to higher temperatures as the cooling rate decreased. Slow cooling decreases both vacancy and solute concentrations in the matrix. It also reduces the uniformity of scattered vacancies in the matrix. Consequently, the time necessary to form the critical mass of solute for GPB zone formation is prolonged.

Whereas little or no GPB zones formed during the DSC scan of air-cooled samples, GPB zone dissolution occurred significantly in both materials. Thus, some GPB zones formed during air cooling [9]. The GPB zone dissolution peak temperatures appear not to have been significantly affected by cooling rate in both materials. However, the peak reaction temperatures for intermediate phase (S' and θ') formation in samples quenched in air, hot water, ethanol, and liquid nitrogen occurred at higher temperatures than that of water. Couper and Polmear [12] have suggested that quenched-in vacancies are required to facilitate nucleation of intermediate S' precipitates. The fact that the peak reaction temperature is less in samples quenched in laboratory water than in those quenched in other media suggests that there has been a significant loss of vacancies in the latter specimens. Although it was difficult to calculate the reaction enthalpies for the formation of the intermediate precipitates in the composite material, a close examination of peak C in both materials reveals that the composite is more quench sensitive than the unreinforced alloy. The reaction enthalpies are much lower in the composite than in the parent alloy quenched in the same medium.

5. Conclusions

1. Cooling rate is a very important factor in the manufacturing and post-fabrication (e.g. solution heat treatment) processes of 2618 aluminum alloy. In particular, it affects the as-quenched and aged properties of this alloy about equally. However, it does not alter the aging sequence of 2618 aluminum and the MMC. That is, the aging sequence is independent of the quenchant used.
2. The unreinforced 2618 aluminum is a quench sensitive alloy. The addition of Al_2O_3 particles increases its quench sensitivity.
3. Both the unreinforced alloy and the MMC show two different types of quench sensitivity behavior. At low cooling rates, where the formation of intermediate precipitates appear to dominate, the MMC is more quench sensitive than the parent alloy due to the lack of quenched-in vacancies required for GPB zone formation. However, at high cooling rates (where quench sensitivity seems to be vacancy and dislocation controlled) the MMC shows a lower sensitivity than the unreinforced alloy.

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