

Communications

Development of Reproducible and Increased Strength Properties in Thick Extrusions of Low-Alloy Al-Zn-Mg-Cu Based AA 7075

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The present article deals with the studies undertaken to resolve an industrial problem, where the desired minimum tensile properties in the T651 temper, *i.e.*, 0.2 pct PS = 530 MPa, UTS = 580 MPa, and elongation (in 50-mm gage length) = 7 pct, were difficult to obtain in a reproducible manner in low-alloy (*i.e.*, alloy containing major alloying elements in amounts not greater than those specified by the nominal compositions in the specification) AA 7075 round bar extrusions having 80-mm diameter.

The weight composition of AA 7075 alloy is Al-(5.1 to 6.1)Zn-(2.1 to 2.9)Mg-(1.2 to 2.0)Cu-(0.10 to 0.28)Cr-0.20Ti, and the maximum limits of Fe, Si, and Mn impurities are 0.5, 0.4, and 0.3 pct, respectively.^[1,2] For the present purposes, the Zn and Mg contents in the alloy were varied from their lower limits to 5.6 and 2.5 pct, respectively, while the upper limit for Cu was restricted to 1.4 wt pct to make the optimum homogenization cycle effective,^[1] to provide the dimensional stability to the final products,^[1] and to minimize the weight variation between the identical parts of a particular component of interest (this is because Cu has a strong tendency to segregate). Furthermore, in order to achieve the specified ductility in the alloy products^[3,4] and to make such alloy products respond suitably to hard anodizing treatments,^[5,6] the contents of Fe, Si, and Mn impurities were restricted to less than 0.15, 0.10, and 0.10 wt pct respectively.

The typical T6/T651 tensile properties of AA 7075 alloy at room temperature are 0.2 pct PS = 500 MPa, UTS = 570 MPa, and elongation (in 50-mm gage length) = 11 pct.^[1,2] These strength levels are, therefore, lower than those required in the extrudates of present interest. In this article, results using transmission electron microscopy (TEM) and tensile tests are presented to show that the use of an artificial aging treatment at about 100 °C for a short duration prior to aging the alloy in the standard temperature range of 120 °C to 135 °C (for the T6/T651 temper) is essential in order to obtain the desired combinations of the tensile properties in low-alloy thick extrusion products of AA 7075 in a reproducible manner. The theoretical basis for the suggestion of the use of the two-step artificial aging treatment is first discussed with reference to the use of similar treatments in the Al industries for 7000 series Al alloys for increasing the stress corrosion cracking (SCC) resistance with reduced loss in strength,^[1,2] and for obtaining higher strength in thick forgings quenched in warm/boiling water.^[1,2]

The chemical composition (wt pct) of the round bar AA 7075 extrusions (having 80-mm diameter) used in the present investigation is Al-5.3Zn-2.2Mg-1.3Cu-0.18Cr-0.04Ti-0.14Fe-0.07Si-0.06Mn. The extrudates were solution treated at 470 °C for 3 hours followed by quenching in water at room temperature. The as-quenched extrudates were controlled stretched (~2 pct for stress relief purposes), naturally aged for 100 hours, and subjected to the artificial aging treatments. For the single-step aging treatment, the aging temperature was 125 °C, and for the two-step artificial aging treatment, the aging temperatures were 100 °C and 125 °C. The optimum aging hours (in terms of maximum hardness achieved) at individual artificial aging temperatures were evaluated by Vickers hardness testing, using a 10-kg load. It was found that 6 hours at 100 °C and 24 hours at 125 °C are the optimum aging times. The tensile properties of the fully heat-treated extrudates were evaluated by tensile tests using the standard round tensile bars (*i.e.*, T 108 tensile specimens) having a 12.5-mm diameter and a 50-mm gage length. The tensile specimens were machined from the longitudinal sections of the extrusions, and the location of sampling was confined to the central portions of the round bar extrusions. Specimens were tested at a crosshead speed of 1 mm/min. All tensile tests were performed at room temperature. The microstructural changes in the alloy were examined by TEM. Specimens for TEM were prepared by electrolytic polishing using 30 pct nitric acid and 70 pct methanol at -35 °C. Thin foils were examined on a PHILIPS* EM 430T electron microscope

*PHILIPS is a trademark of Philips Electronic Instruments Corp., Mahwah, NJ.

operating at 150 kV.

The theoretical basis for the use of two-step aging treatment for 7000 series Al alloys is as follows. A common approach to obtain high strength in precipitation hardening systems is to increase the solute supersaturation, thus increasing the volume fraction of the strengthening precipitates that form during aging. The reported results in the literature on 7000 series Al alloys, however, suggest that the relationships between the alloy contents and the corresponding strength properties (obtained upon heat treatment) are not always linear.^[1,2,4,7,8] The implication is that for a given heat treatment cycle, lower strength values may be obtained in the alloy despite the presence of the major alloying elements in relatively higher amounts. This unpredictable variation in the strength properties as functions of alloy compositions is understood to be due to the critical need of a high supersaturation of free vacancies during quenching for the nucleation of strengthening precipitates, *i.e.*, stable Guinier-Preston G-P zones and metastable MgZn₂ (η') phase, in this alloy series.^[9-12] This is further discussed as follows.

The decomposition of the supersaturated solid solution of ternary Al-Zn-Mg based alloys after solution treatment and quenching starts with the formation of spherical and fully coherent solute clusters, *i.e.*, G-P zones.^[1-3,11-13] In ternary alloys, such G-P zones contain both zinc and magnesium.^[14,15] In quaternary Al-Zn-Mg-Cu alloys, these zones may also contain Cu, and Cu has a decisive effect of nucleating and stabilizing the early stages of G-P zone formation.^[16] During natural aging, the spherical G-P zones

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nucleate and grow.^[11] Numerous experimental results point toward the fact that the initial 100 hours of natural aging is crucial in terms of rapid kinetics involving nucleation of G–P zones containing both Zn and Mg, and that the delay between quenching and artificial aging (t) in the time interval of 3 hours $< t < 100$ hours adversely affects the strength properties of the alloy obtained upon artificial aging.^[11] During artificial aging, the G–P zones dissolve partially or completely depending on whether the G–P zones could attain a critical size,^[17,18] a critical composition involving Mg and Zn,^[14,15] and/or whether the aging temperature is below or above the G–P zone solvus.^[17,18] Recent studies have confirmed that the formation of stable G–P zones in these alloys critically depends upon a high degree of free vacancy supersaturation during quenching.^[11] During artificial aging, the stable G–P zones act as the nucleation centers for the metastable MgZn₂ (η') phase.^[3,10,11,17,18] Any alteration in the free vacancy concentration in solution during quenching would, therefore, have a marked influence on the precipitation processes in these alloys. The hardening ability of these alloys accordingly depends on both the solutionizing and the quench bath temperature, as well as on the cooling rate.

Yet another major factor influencing the strength properties of these series of alloys is the thickness of the product. For a given product, as its thickness increases, the differences in the cooling rates at which the surface and the interior of the product get quenched become progressively higher. Consequently, for relatively thick products, the precipitation kinetics would not be the same across the product section. The result would be the reduced driving force for the desired fine and uniform precipitation at the center compared to that at the midradius or the surface regions of the products. This is owing to the loss of excess vacancies, as well as solutes (because, besides the loss of vacancies and solutes through migration of vacancies and solute-vacancy complexes to the grain boundary sinks, the solutes consumed during slow cooling in the process of formation of coarse precipitates at heterogeneous nucleation sites such as dislocations, interphase boundaries, *etc.* would also not be available for the formation of desired fine and uniform distribution of strengthening precipitates in the matrix during artificial aging), due in turn to the relatively slow rate of cooling attained by the center region of the product. The desired response to aging can, however, be restored in such alloy products by the use of two-step artificial aging treatment.

The use of two-step (/multistep) artificial aging treatment, involving aging below the G–P zone solvus for several hours followed by aging above the G–P zone solvus for a shorter period, for enhancing the SCC resistance of this series of alloys has been well known.^[1,2,19] This type of aging practice is attractive, because the SCC resistance is increased without much loss in the strength of the alloy. During the first-step aging, the G–P zones nucleate and grow; the equilibrium vacancies contribute toward the stability of such G–P zones *via* changes in their chemistry as well as size. During the second-step aging, the stable G–P zones aid the nucleation of the η' -phase precipitates. Typically, the first- and second-step aging temperature ranges for the T7X type treatments are 100 °C to 120 °C and 160 °C to 170 °C, respectively.^[1,2] For the T6/T651 temper of

Table I. Influence of Two-Step Artificial Aging Treatment on the Longitudinal Tensile Properties of the Extrusions

| Artificial Aging (Time and Temperature) | Tensile Properties | | Pct Elongation (in 50-mm Gage Length) |
|---|--------------------|------------|---|
| | 0.2 PS (MPa) | UTS (MPa) | |
| 24 h at 125 °C | 507 to 515 | 560 to 579 | 9 to 10 pct |
| 6 h at 100 °C + 24 h at 125 °C | 548 to 556 | 597 to 610 | 9 to 10 pct |

present interest, on the other hand, the recommended single-step aging temperature lies in the range of 120 °C to 135 °C.^[1,2] Further, the peak-aged microstructure in the T6/T651 temper of present interest is known to contain both stable G–P zones and metastable MgZn₂ (η') phase.^[20,21] The implication is that the formation of the strengthening η' phase in the T6/T651 temper can also be aided by the prior formation of the stable G–P zones in the microstructure, and this, in turn, can be ensured by the use of a prior low-temperature aging at about 100 °C; the selection of this temperature is made on the basis that it is below the G–P zone solvus for the present series of alloys.^[22] It is notable that for thick forgings, which are quenched in warm/boiling water to avoid the problem of quenching stress, the use of an initial low-temperature aging at 100 °C is recommended in order to promote the response to aging.^[1,2] The results presented later in this article aim at demonstrating that the introduction of this low-temperature aging is important for the thick products, which are quenched in water even at the ambient temperature.

In order to examine the influence of two-step artificial aging treatment on the tensile properties of the present alloy, the extrudates were solution treated, quenched in water at room temperature, controlled stretched 2 pct, naturally aged for 100 hours, and subsequently artificially aged at (a) 125 °C for 24 hours and (b) 100 °C for 6 hours followed by aging at 125 °C for 24 hours. Several batches of the present materials were subjected to such heat treatments and subsequent tensile tests to check the level of reproducibility of the mechanical properties. The tensile results presented in Table 1 show that the introduction of the prior low-temperature aging at 100 °C noticeably increases the strength properties of the extrudates with no adverse effect on the ductility.

Figures 1(a) through (c) represent the transmission electron micrographs of the alloy artificially aged for (a) 6 hours at 100 °C, (b) 24 hours at 125 °C, and (c) 6 hours at 100 °C followed by 24 hours at 125 °C, respectively. Figure 1(a), imaged in $\langle 110 \rangle_{Al}$ orientation, shows a fine and uniform distribution of spherical G–P zones in the alloy. The selected area electron diffraction (SAED) pattern obtained from Figure 1(a) is shown in Figure 1(d). The pattern shows diffraction spots due only to the matrix Al, suggesting that the fine precipitates shown in Figure 1(a) do represent G–P zones having the same crystal structure as that of the matrix. Figure 1(e) represents an SAED pattern obtained from Figure 1(c). The pattern shows the presence of streaks along the $\langle 111 \rangle_{Al}$ direction, suggesting the presence of η' plates on the $\{111\}_{Al}$ matrix planes. The presence of $1/3\langle 220 \rangle_{Al}$ and $2/3\langle 220 \rangle_{Al}$ spots in the diffraction pattern, as shown in Figure 1(e), are also consistent with the presence of η' in the microstructure.^[20] The SAED pattern obtained

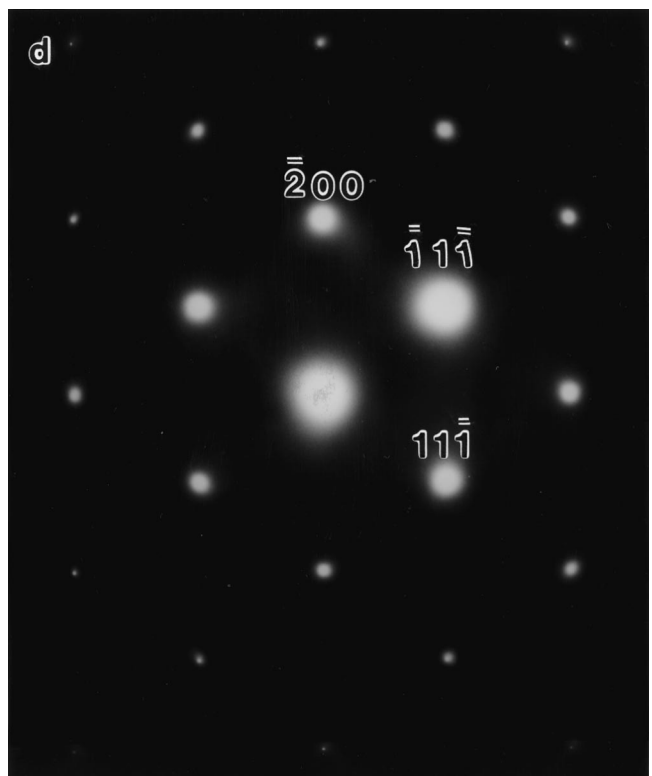
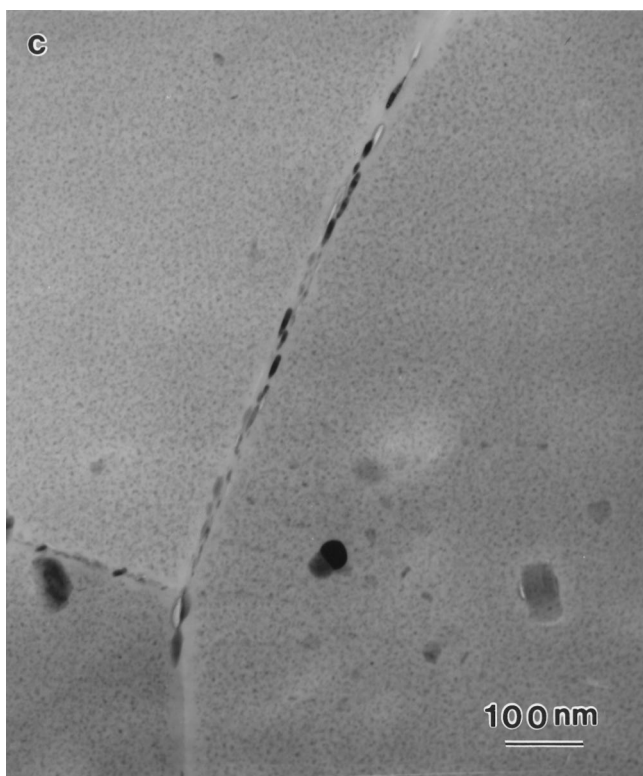
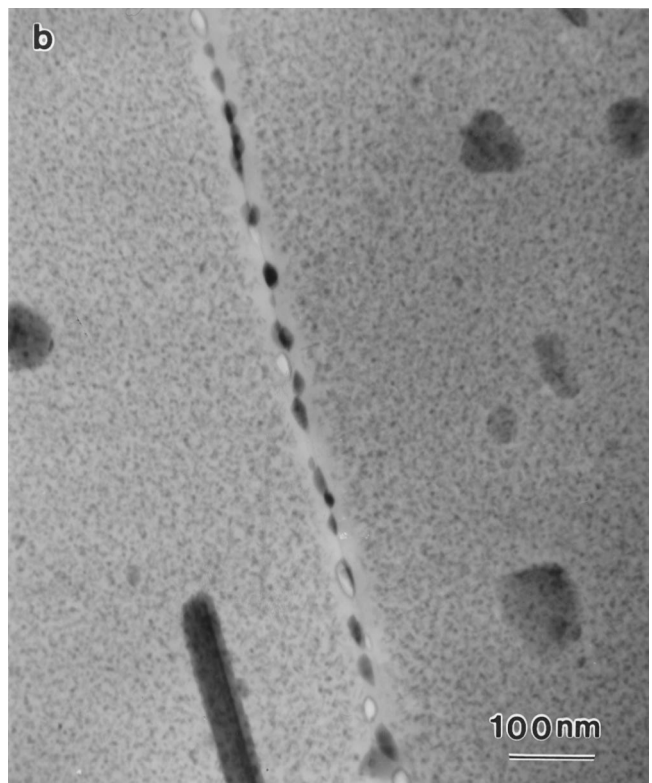
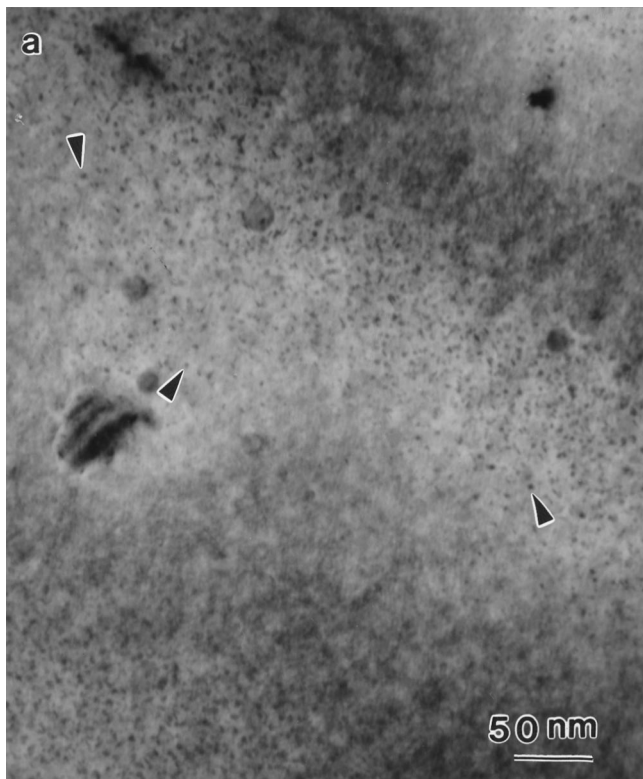


Fig. 1—Bright-field images showing development of microstructure in the alloy when solution treated, water quenched, controlled stretched 2 pct, naturally aged for 100 h, and artificially aged at (a) 100 °C for 6 h, (b) 125 °C for 24 h, and (c) 100 °C for 6 h followed by aging at 125 °C for 24 h. Electron beam is approximately parallel to $\langle 110 \rangle_{Al}$ zone in each case. (d) represents a SAED pattern corresponding to (a). Arrows in (a) point out the presence of spherical G-P zones.

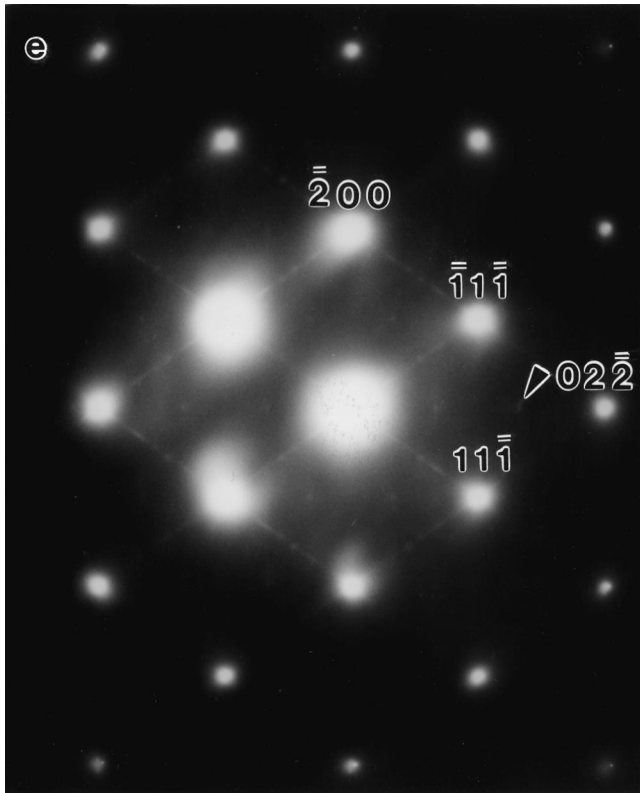


Fig. 1 Continued—(e) represents a SAED pattern corresponding to (c). The arrow in (e) points out the presence of $2/3\langle 220 \rangle_{Al}$ reflection.

from Figure 1(b) was also examined and was found to produce diffraction features identical to that shown in Figure 1(e). Figure 2 represents a dark-field micrograph (imaged using the $2/3\langle 220 \rangle_{Al}$ reflection, as shown in Figure 1(e)) of the alloy subjected to the two-step aging treatment. The micrograph shows the presence of numerous small plate-shaped η' precipitates. The present results are, therefore, consistent with the earlier results that η' is the major phase in this series of alloys heat treated to the T6/T651 temper.^[20,21]

Comparison of Figure 1(b) with (c) suggests that a reduction in the size of the second-phase precipitates, formed in the matrix, has been brought about by the introduction of the prior low-temperature aging at 100 °C. This is consistent with the increase in the strength of the alloy subjected to the two-step artificial aging. Further examination of the microstructures shown in (b) and (c), together with the examination of similar microstructures in several other thin foils of the similarly treated materials, revealed that the use of the prior low-temperature aging reduces both the average width of the precipitate free zones (PFZs) adjacent to the grain boundary (by about 20 pct), as well as the average diameter of the grain boundary precipitates (by about 25 pct). The consequence of these changes is that the ductility remains unchanged despite the observed increase in the strength properties of the material. It is further pointed out that the simultaneous reduction in both the width of the PFZs adjacent to the grain boundary and the size of the grain boundary particles must be a direct consequence of the formation of an increased number of stable

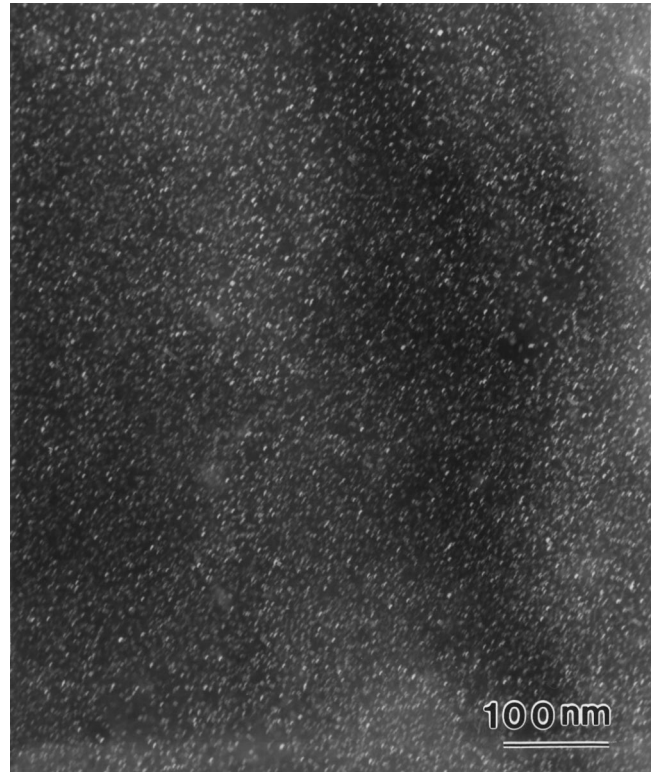


Fig. 2—Dark-field image (using $2/3\langle 220 \rangle_{Al}$ reflection arrowed in Fig. 1(e)) showing the presence of fine η' plates in the alloy subjected to the two-step artificial aging treatment.

G–P zones in the alloy during the low-temperature (*i.e.*, the first-step) artificial aging treatment. The use of the initial low-temperature artificial aging, therefore, prevents loss of solutes to grain boundary (by forming stable G–P zones and, thereby, preventing their dissolution at the second-step artificial aging temperature), leading to higher solute concentration in the matrix, which in turn contributes toward the formation of desired strengthening precipitates.

The present results that the use of the two-step artificial aging treatment enables higher strength properties to be achieved in the alloy with reduced Zn and Mg contents has an additional advantage in that the susceptibility to SCC is known to reduce with decreasing Zn + Mg contents in this alloy series.^[1,2] This, together with the previously documented results that the strength properties of these materials subjected to the two-step artificial aging treatments (employed for increasing the SCC resistance) are not affected by the time delay between quenching and aging,^[1] should make the use of the two-step artificial aging treatment commercially more attractive compared to the single-step artificial aging treatment used for the T6/T651 temper.

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Discussion of “Kinetics and Phase Transformation Evaluation of Fe-Zn-Al Mechanically Alloyed Phases”*

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In a recent contribution, Uwakweh and Liu reported some interesting results on the formation of ternary Zn-Fe-Al compounds through a mechanical alloying route. As correctly pointed out by the authors, galvanizing processes are (frequently) nonequilibrium in nature, so it is important to understand the alloy formation mechanisms for efficient and predictable applications of Zn coatings. The information reported by the authors is certainly of interest to the galvanizing industry.

However, it appears that the authors were not aware of many new developments in this field at the time when their work was analyzed and summarized for publication. It was

already firmly established that the Γ' (or Γ_1 in the authors' notation) phase is an equilibrium phase in the Zn-Fe-Al ternary system.^[1,2,3] It was found that δ phase produced in molten galvanizing alloys is metastable at temperatures below 450 °C, and it transforms to Γ' after long-term annealing.^[1,2,3] The phase relation in the Zn corner of the Zn-Fe-Al system at temperatures relevant to galvanizing processes has been recently summarized in the 450 °C isotherm by Tang.^[4] According to this most up-to-date isotherm, or the partial 440 °C and 470 °C isotherms proposed by Yamaguchi *et al.*,^[2] all the alloys studied by Uwakweh and Liu should contain a certain amount of the Γ' phase. However, these two researchers failed to detect this phase in their samples. This is believed to be due to two reasons. First, the heat-treatment time, 3 hours, as mentioned in the article, was probably insufficient for the establishment of an equilibrium state, especially at temperatures below 400 °C. Second, only X-ray diffraction (XRD) was employed for the identification of the constituents of all the alloys. It is very difficult to distinguish the δ and Γ' phases using only the XRD technique. These two phases, due to their complex crystal structures, produce a large number of overlapping XRD peaks. Their XRD patterns are similar, and the differences are subtle. According to Cook and Grant,^[5] the only differentiating peak is located at $2\theta = 41.4$ deg, which is unique to the Γ' phase. However, this peak can be easily overlooked because the δ phase has a strong peak at $2\theta = 41.6$ deg. Re-examination of the patterns published by the authors, as shown in Figures 9(c) and (d) indicated that the peak unique to the Γ' phase did exist. Also, the peak indexed as $(101)_\delta$ by the authors was actually generated by $(660)_{\Gamma'}$ and $(822)_{\Gamma'}$. This is the strongest peak in the Γ' phase's spectra. The three peaks, which the authors assigned to the FeAl_3 phase, as well as the unattributed peak all belong to the Γ' phase. The peak corresponding to a d -spacing of 2.18 Å was actually the telltale peak mentioned earlier. It was generated by $(820)_{\Gamma'}$ and $(644)_{\Gamma'}$ planes. It appears that the indexing of the peaks carried out by the two authors was slightly oversimplified. For example, if the peak identified as $(101)_\delta$ by the authors were generated by the δ phase, at least four planes of the δ phase would have contributed to it, according to Cook and Grant.^[5]

The aforementioned problems are, in part, caused by the isotherms used by the authors. The phase diagrams^[6,7] cited in the article are not the best ones available in the open literature. The 350 °C isotherm used by the authors is schematic and incomplete. This problem is easily recognized because most intermetallic compounds are stable at temperatures below 400 °C, and the remaining invariant reactions in the system occur in the region close to the binary Zn-Al side. Consequently, the 350 °C isotherm should very much resemble the 400 °C isotherm. In fact, it did not contribute to the interpretation of the research results and only caused confusion. The authors were misled by this incomplete isotherm and believed that their alloys at temperatures below 350 °C should contain $\alpha + \eta + \text{FeAl}_3$ (misprinted throughout the paper as Fe_3Al), as shown in Table II of the article. Actually, if equilibrium is established, the alloys should all contain the Γ' phase, but not the FeAl_3 phase.

To date, the most comprehensive and accurate constitutional and chemical study of the Zn-Fe-Al ternary system was still the one carried out by Köster and Gödecke close

*O.N.C. UWAKWEH and Z. LIU: *Metall. Mater. Trans. A*, 1997, vol. 28A, pp. 517–25.

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