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LETTER

Orientation relationships and morphology of S phase in friction stir welded Al–Cu–Mg alloy

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It is generally believed that S phase has lath-morphology. Both lath and rod-shaped S phase were observed in the present study and a closer observation of the selected area diffraction patterns (SADPs) revealed an interesting difference in their orientation relationship with the matrix. A modified orientation relationship has been recently discovered and related to the S phase morpholgy. Although at this time, the effect of the orientation relationship and morphology of S phase on the mechanical properties of Al–Cu–Mg alloys is not understood, it is worthwhile to investigate this observation for increasing the understanding of S phase precipitation and growth. Although there have been several investigations relating to the microstructure of friction stir welds in Al–Cu–Mg alloys [1–4], precipitation and growth of S phase in these welds have not been adequately addressed.

Two friction stir welds produced on 1-mm thick 2024-T3 commercial aluminum alloy (Al–4.4Cu–1.5Mg–0.5Mn) using different heat inputs were studied using transmission electron microscopy (TEM) operating at 200 kV. The process conditions of these welds are shown in Table 1. The ratio of tool rotation speed and the traverse rate gives the heat index *N*/*v*. The tool used for friction stir welding consisted of a shoulder with a diameter of 7 mm and a 1 mm long pin having a diameter of 2.5 mm. Based on the process parameters and the tool dimensions, it can be calculated that the heat input per unit length for the low heat input weld and high heat input weld were 99.8 and 269.8 kJ/mm, respectively. This suggests that the high heat input weld experienced significantly higher peak temperature as compared to the low heat input weld.

The location in the heat affected zone (HAZ) just outside the thermomechanically affected zone exhibited coarse S phase, which had precipitated due to the heat of friction stir welding. Figure 1 shows the bright field (BF) images and SADPs from these regions for both welds. The majority of S phase in the low heat input weld (1) exhibited a rod morphology, while the S phase in the high heat input weld (2) exhibited a lath morphology. It should be noted that in the [001] SADP for the low heat input weld, both $\{112\}_S$ and $\{131\}_S$ reflections were strong, while in case of the high heat input welds, only the $\{131\}_S$ reflections were strong.

S phase is generally described as laths, which grow on the $\{021\}_{Al}$ planes, elongated in $[001]_{Al}$ direction. The accepted crystallographic orientation relationship between the S phase and the aluminum matrix, as proposed by Bagaratskii [5, 6], Silcock [7] and many others is as follows:

$$[100]_S \parallel [100]_{Al}, [010]_S \parallel [021]_{Al}, [001]_S \parallel [012]_{Al}. \qquad (1)$$

The orientation relationship is schematically shown in Fig. 2a [8]. The angle between $[010]_{Al}$ and $[021]_{Al}$ is 26.56°, which is also the angle between $[010]_S$ and $[010]_{Al}$. The angle between $[02\bar{1}]_S$ and $[010]_S$ is found to be 21.145° and that between $[013]_S$ and $[010]_S$ is found to be 66.68° using the formula for angle between directions for orthorhombic crystal structure and using lattice parameters: $a = 0.4$ nm, $b = 0.923$ nm, and $c = 0.714$ nm. Thus, the angle between $[02\bar{1}]_S$ and $[010]_{Al}$ is 5.415° and that between $[013]_S$ and $[010]_S$ is 3.24°. As a result, we will get a stronger reflection of $(11\bar{3})_S$ from the $[013]_S$ zone axis in the $[001]_{Al}$ SADP, than, for example, a $(112)_S$ reflection since the $[02\bar{1}]_S$ is about 5.5°

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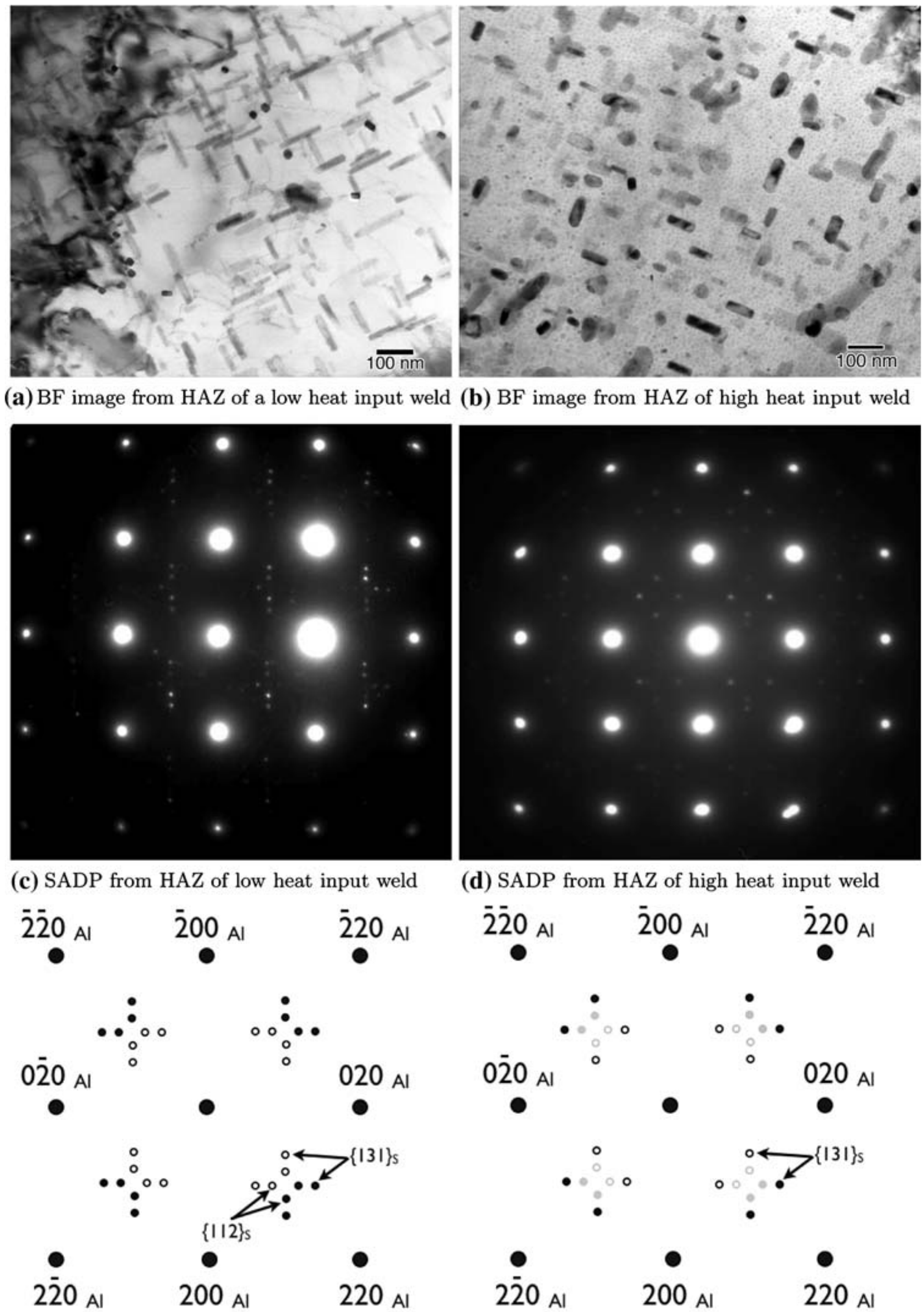
Table 1 Friction stir welding process parameters

Weld	Tool rotation speed (RPM)	Traverse rate (mm/min)	Shoulder plunge depth (mm)	*N*/*v* (rev/mm)
1	2,500	130	0.09	19.2
2	2,600	50	0.09	52

away from $[010]_{Al}$. (The presence of $\{131\}_S$ reflections in spite of the fact that $[013]_S$ is not parallel to $[001]_{Al}$ can be explained by the large radius of the Ewald sphere, which would touch the points in the reciprocal lattice although they do not satisfy Bragg law). This explains the fact why strong {131} reflections are observed, and $\{112\}_S$ reflections are either absent or weak in Fig. 1d.



(a) BF image from HAZ of a low heat input weld (b) BF image from HAZ of high heat input weld

(c) SADP from HAZ of low heat input weld (d) SADP from HAZ of high heat input weld

(e) Schematic SADP from HAZ of low heat input weld (f) Schematic SADP from HAZ of high heat input weld

Fig. 1 TEM BF images and SADPs showing S phase in the HAZ of high and low heat input welds

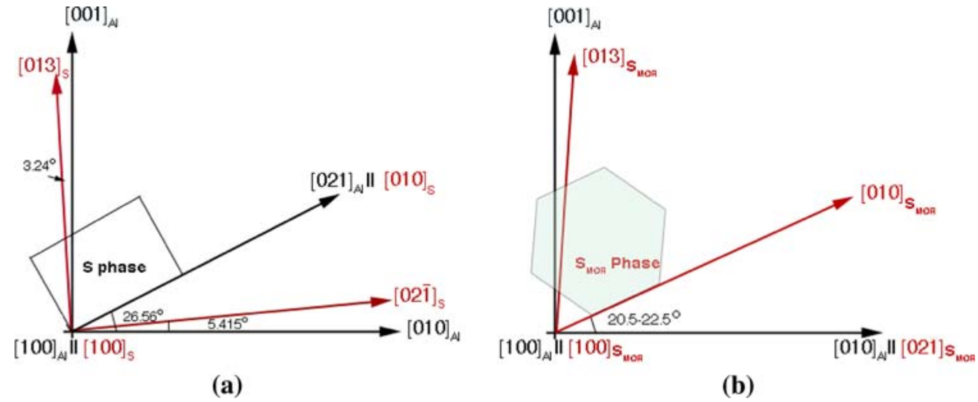


Fig. 2 Classical (**a**) and modified (**b**) orientation relationships between S phase and the aluminum matrix (adapted from [8])

Recently, several investigators [8–10] have reported a modified orientation relationship between S phase and the aluminum matrix. Radmilovic et al. [9] studied the structure and morphology in an Al–2.01Cu–1.06Mg–0.14Zr–0.08Fe (wt%) alloy and observed two types of precipitates, one with planar interfaces called type I and one with a stepped interface called type II. They observed that there was a difference of approximately 5° between the orientation relationships of both types. The following orientation relationship was proposed by the Radmilovic et al. based on the angle measurements of the Fourier transforms of high-resolution TEM (HRTEM) images:

$$[100]_S \parallel [100]_{Al}, [010]_S \parallel [01\bar{2}]_{Al}, [002]_S \parallel 1/2[073]_{Al}. \tag{2}$$

Majimel et al. studied the orientation relationships and morphology in an Al–2.68Cu–1.86Mg–0.21Fe–0.21Si–0.34Mn–0.24Ni–0.09Ti (wt%) alloy and they also observed two types of S phase, and proposed the following orientation relationship for the type II precipitate based on the angle measurements of the Fourier transforms of HRTEM images:

$$[100]_S \parallel [100]_{Al}, [010]_S \parallel [025]_{Al}, [001]_S \parallel [0\bar{5}2]_{Al}. \tag{3}$$

They observed that the type II precipitates were misoriented by 4.84°.

Kovarik et al. [8] studied the orientation relationship and morphology of S phase in an Al–2.96Mg–0.42Cu–0.12Si–0.25Mn–0.21Fe–0.007Zn–0.002Ti alloy. They also observed a modified orientation relationship in the S phase. They observed that the orientation relationship was modified by 4°–6° from the classic orientation relationship described by Eq. 1. It should be noted that Kovarik et al. suggested that the "type II" S phase be called S″ in accordance with the notation initially used by Bagariatskii; however, in order to avoid confusion, here they are simply referred to as S phase with modified OR (S_{MOR}) hereafter. As shown in Fig. 2b, in case of S_{MOR}, $[02\bar{1}]_S$ is rotated such that it coincides with $[010]_{Al}$. As a result, for S_{MOR} the $\{112\}_S$ reflections would be strong. The $\{131\}_S$ type reflections are not affected because although $[013]_S$ is also rotated by about 5°, the net angle between $[013]_S$ and $[001]_{Al}$ does not change appreciably, only the sign changes. The morphology of S_{MOR} observed by both Kovarik et al. and Majimel et al. suggested that these were rods. Majimel et al. plotted the width (L) of the planar, coherent interface between S and the aluminum matrix (the $(001)_S \parallel (021)_{Al}$ interface) against the angular misorientation and found that most of the precipitates with smaller value of L exhibited larger misorientation. (A precipitate with smaller L would represent a rod, while one with a larger L would represent a lath). These observations suggest that S_{MOR} is usually formed as rods.

It should be noted that the $\{112\}_S$ reflections were strong in case of the low heat input weld and also they were rod shaped; thus, they had a modified orientation relationship. Thus, the observations in this study completely support the findings of the above investigators. It is interesting to note that rod-shaped S_{MOR} (and some lath-shaped S) was formed in the low heat input weld, while only lath-shaped S phase was formed in the high heat input weld, which indicates the role of temperature on the morphology and orientation relationship of the precipitated S phase. The reason for this precipitation behavior is not clearly understood; however, it is believed that it is related to the number of coherent interfaces in both types of S phase. The change in free energy for heterogeneous nucleation is given by [11]:

$$\Delta G_{het} = -V(\Delta G_v - \Delta G_s) + A\gamma - \Delta G_d \tag{4}$$

where $V\Delta G_v$ is the change in free energy due to formation of a new phase with volume V, $A\gamma$ the increase in free energy due to formation of an interface with an area of A, $V\Delta G_s$ the increase in free energy due to the misfit strain produced due to the formation of the new phase in the matrix, and ΔG_d the decrease in free energy related to nucleation of the precipitate on a defect like dislocations, boundaries, vacancies, etc. If the interface is coherent, the critical size of the nucleus required for precipitation can be lower, because it will have a low-energy interface and the term $A\gamma$ would be smaller. In other words, higher the number of coherent interfaces, easier the nucleation. The S_{MOR} has two coherent interfaces: the well-known $(001)_S \parallel (021)_{Al}$ interface and another, recently discovered, $(021)_S \parallel (014)_{Al}$ interface [8]. Thus, thermodynamically, it is easier for Al_2CuMg to nucleate as S_{MOR} rather than the S phase with the classic orientation relationship at relatively lower temperatures, which explains the formation of S_{MOR} in the low heat input weld. As the precipitate grows, the lattice strain would increase, preventing the $(014)_S$ interface to remain coherent, since it is difficult for this interface to maintain coherency due to the presence of a higher misfit and it eventually becomes incoherent, allowing the growth normal to this interface. Thus, the rod-shaped S phase would transform to lath-shaped S phase with the rotation of S phase to the classic orientation relationship, which is stabler than S_{MOR}, which explains the observation of some lath-shaped S phase in the low heat input weld. For the high heat input weld, due to the presence of higher temperature, this transformation would begin early during it's growth, resulting in mainly lath-shaped S phase with classic orientation relationship.

The higher length of S phase observed in low heat input weld as compared to the high heat input weld can be explained simply by the classical theory of precipitation, according to which a precipitate with a smaller radius would have a higher velocity of growth than one with a larger radius. Since rods have a smaller radius at their incoherent, ends, they grow faster and hence the S phase in the low heat input weld are longer in the $[001]_{Al}$ direction.

In summary, two types of orientation relationships between S phase and the aluminum matrix were observed, based on the process parameters used. A relation between the orientation relationships and the morphology of S phase was also observed. In the low heat input weld produced at 2,500 RPM and 130 mm/min, S phase exhibiting modified orientation relationship and principally rod morphology was observed. In the high heat input weld produced at 2,600 RPM and 50 mm/min, S phase exhibiting a classical orientation relationship and principally lath morphology was observed. These observations can be related to the thermodynamics of precipitation of coherent phases in Al–Cu–Mg alloys.

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