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Effects of Sc and Zr microalloying on the microstructure and mechanical properties of high Cu content 7xxx Al alloy

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Abstract: The effects of Sc and Zr microalloying on the microstructure and mechanical properties of a 7xxx Al alloy with high Cu content (7055) during casting, deformation, and heat treatment were investigated. The addition of Sc and Zr not only refined the grains but also transformed the θ -phase into the W-phase in the 7055 alloy. Minor Sc and Zr additions enhanced the hardness and yield strength of the 7055-T6 alloy by strengthening the grain boundaries and Al₃(Sc,Zr) precipitates. However, a further increase in the Sc and Zr fractions did not refine the grains but instead resulted in the formation of the large-sized W-phase and primary coarse Al₃(Sc,Zr) phase and subsequently deteriorated the mechanical properties of the alloys. The 7055 alloy with 0.25Sc addition exhibited the best mechanical property among the prepared alloys.

Keywords: aluminum alloys; microstructural evolution; mechanical property; grain refinement

1. Introduction

The addition of Sc and Zr into Al alloys results in the formation of a complex $Al_3(Sc,Zr)$ phase [1-3]. The primary $Al_3(Sc,Zr)$ phase several microns in size can promote heterogeneous nucleation in Al melts during solidification and subsequently refine the grains of Al castings [4]. The nanosized $Al_3(Sc,Zr)$ phase that precipitates during a high-temperature homogenization treatment can further refine the grains of Al alloys during subsequent heat treatments and deformation because of its pinning effect on grain boundaries (GBs) [5–13]. Thus, Al alloys with Sc and Zr added have attracted considerable attention because of their fine-grained microstructures and high strength.

The 7xxx series (Al–Zn–Mg(–Cu)) alloys are widely used in the aircraft manufacturing industry because of their excellent mechanical properties, low densities, good formability, and high fracture toughness [14–17]. Adding Sc and Zr effectively improves the mechanical properties of 7xxx alloys with low Cu contents (mass ratio of Cu in the alloy less than 0.8%) through grain refinement [4,6–9]. The 2xxx series (Al-Cu base) alloys also exhibit high strength [18-20], and grain refinement induced by the addition of Sc has also been observed in these alloys [21-26]. Chen et al. [27] and Jiang et al. [28] found that the segregation of Sc atoms at the Al/ θ' -phase interface inhibits the formation of large θ -phases (Al₂Cu) in Al-2.5wt% Cu alloys during aging. Thus, the mechanical properties of the 2xxx series alloys can also be improved by the addition of Sc. However, when the Cu content in the Al-Cu base alloy with Sc exceeds 5wt%, Sc dissolves into the θ -phase and induces a phase to transformation to the W-phase (AlCuSc) during the homogenization process [24-25]; the W-phase adversely affects the strength and plasticity of Al alloys. Furthermore, the formation of the W-phase consumes Sc atoms, thereby decreasing the amounts of Al₃Sc or Al₃(Sc,Zr) phases present in the alloys. Thus, the strengthening effect of Sc addition in Al-Cu systems is not as remarkable as that observed in the 7xxx alloys.

Recently, 7xxx alloys with high Cu contents (higher

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than 2wt%) have been developed. These alloys exhibit higher strength and greater ductility than traditional 7xxx Al alloys [29–32]. However, the interaction between Sc and Cu in 7xxx Al alloys with high Cu contents has not been systematically investigated, and the effects of Sc and Zr microalloying on the mechanical properties of these alloys have not been determined.

In the present study, the effects of various Sc and Zr contents on the microstructure of an Al–Zn–Mg–Cu alloy (7055) with Cu content as high as 2.2wt% under casting, homogenization, rolling, and T6 treatment conditions are investigated. The mechanical properties of the 7055 alloy and 7055 alloy with different Zr and Sc additions (7055–xZr–ySc) in the T6 state are subsequently examined. The results of this study provide a basis for the compositional design of high-performance Al alloys.

2. Experimental

Ingots (7055 and 7055–xZr-ySc) were fabricated by casting. Their chemical compositions are shown in Table 1. The ingots were homogenized at 470°C for 26 h and rapidly quenched to room temperature by immersion in water. The ingots were then subjected to seven passes of rolling from 10 mm to 3 mm at 470°C. The samples were reheated at the prescribed rolling temperature for 3 min between passes. The rolled plates were subsequently subjected to solid-solution treatment at 470°C for 2 h and then aged at 120°C for 1 h to 60 h.

Fable 1.	Composition of 7	7055–xZr–ySc ingots	wt%
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7055–xZr–ySc	Zn	Mg	Cu	Zr	Sc
7055	7.82	1.95	2.24	0.16	-
7055–0.2Sc	7.80	1.94	2.24	0.16	0.2
7055–0.25Sc	7.81	1.93	2.24	0.16	0.25
7055-0.14Zr-0.15Sc	7.82	1.93	2.24	0.30	0.15
7055-0.14Zr-0.2Sc	7.82	1.95	2.24	0.30	0.20
7055-0.24Zr-0.15Sc	7.81	1.95	2.23	0.40	0.15

The microstructure of the alloys were examined by optical microscopy (OM, Leica-DMi8), scanning electron microscopy (SEM, S4800), transmission electron microscopy (TEM, JEOL-2010), and scanning transmission electron microscopy (STEM, F200X). The specimens for OM, SEM, TEM, and STEM were prepared in the rolling direction–normal direction plane of the samples. The TEM and STEM specimens were prepared by grinding to 50 μ m, followed by thinning with a twinjet electropolishing apparatus in a solution of 90vol% methanol and 10vol% acetic acid at −10°C.

Vickers microhardness was measured with an EVE-RONE VH-5 hardness tester. The tensile properties of the samples were evaluated using an Instron 3369 testing machine. The tensile specimens were machined along the rolling direction; the tensile strain rate was 4×10^{-4} s⁻¹. The tensile specimens are illustrated in Fig. 1. Each alloy was tested three times.



Fig. 1. Schematic of a tensile specimen (Unit: mm).

3. Results and discussion

Fig. 2 shows the OM images of the as-cast 7055 and 7055-xZr-ySc ingots. The grain morphologies of these ingots are listed in Table 2. A dendritic structure was observed in the 7055 ingot (Fig. 2(a)). As shown in Figs. 2(b) and 2(c), the addition of 0.2wt% or 0.25wt% Sc promoted the formation of equiaxed grains during casting, and both dendritic and equiaxed grains were observed in the 7055–0.2Sc and 7055–0.2Sc ingots. Only equiaxed grains were observed in the 7055–xZr–ySc ingots when the total mass fraction of Sc and Zr exceeded 0.45 (Figs. 2(d)–2(f) and Table 2).

The primary Al₃(Sc,Zr) phase promotes heterogeneous nucleation of the Al grains, thereby promoting grain refinement [6]. The density of heterogeneous nucleation sites increased with increasing the total mass fraction of Sc and Zr. Thus, refined and equiaxed grains were observed in the 7055-xZr-ySc ingots with high Sc and Zr additions.

Fig. 3 shows OM images of the 7055 and 7055–xZr-ySc ingots after homogenization. The dendritic grains in the 7055, 7055–0.2Sc, and 7055–0.2SSc ingots changed into equiaxed grains after homogenization (Figs. 3(a)–3(c)). The grain structure and size of the 7055–xZr-ySc ingots with high total mass fraction of Sc and Zr did not substantially change after homogenization (Figs. 3(d)–3(f)).

Fig. 4 shows the effect of Sc and Zr addition on the grain size of the homogenization-treated 7055 and 7055-xZr-ySc ingots. The grains of the 7055-xZr-ySc ingots were much finer than those of the 7055 ingot, and the average grain size of the homogenized 7055-xZr-ySc ingots decreased with increasing total mass fraction of Sc and Zr. No further decrease was observed when the total mass fraction of Sc and Zr exceeded 0.5.



Fig. 2. OM images of as-cast ingots: (a) 7055; (b) 7055–0.2Sc; (c) 7055–0.2Sc; (d) 7055–0.14Zr–0.15Sc; (e) 7055–0.14Zr–0.2Sc; (f) 7055–0.24Zr–0.15Sc.

 Table 2.
 Grain morphologies of as-cast 7055–xZr–ySc ingots

7055– <i>x</i> Zr– <i>y</i> Sc	Total mass fraction of Sc and Zr / wt%	Grain morphology
7055	0.16	Dendritic
7055–0.2Sc	0.36	Dendritic, Equiaxed
7055–0.25Sc	0.41	Dendritic, Equiaxed
7055–0.14Zr–0.15Sc	0.45	Equiaxed
7055–0.14Zr–0.2Sc	0.50	Equiaxed
7055-0.24Zr-0.15Sc	0.55	Equiaxed

TEM bright-field (BF) images of the 7055 and 7055–0.25Sc ingots after homogenization are shown in Fig. 5. Nanosized particles, which were confirmed to be the Al₃Zr phase in the 7055 after heat treatment [29–32], were observed in the TEM BF images (Fig. 5(a)). The nanosized particles of the homogenized 7055–0.25Sc ingot were dens-

er than those of the 7055 ingot after the same process (Fig. 5(b)). The chemical composition and selected-area electron diffraction (SAED) patterns indicated that the particles in the homogenization-treated 7055–0.25Sc ingot were the $Al_3(Sc,Zr)$ phase and had the same size and structure as the Al_3Zr particles in the 7055 alloy.

The homogenization treatment led to the precipitation of the nanosized Al₃(Sc,Zr) phase in the Al ingot. The particles with a GB-pinning effect effectively prevented extensive grain growth in the Sc-containing Al ingots during long heat treatment at high temperature. The density of the nanosized Al₃(Sc,Zr) particles increased with increasing total mass fraction of Sc and Zr. Consequently, the grain size of the homogenized 7055–xZr–ySc ingots decreased with increasing total mass fraction of Sc and Zr. No further grain size decrease was observed when the total mass fraction of Sc and Zr was 0.5wt% or greater.



Fig. 3. OM images of homogenized ingots: (a) 7055; (b) 7055–0.2Sc; (c) 7055–0.2Sc; (d) 7055–0.14Zr–0.15Sc; (e) 7055–0.14Zr–0.2Sc; (f) 7055–0.24Zr–0.15Sc.



Fig. 4. Effect of total mass fraction of Sc and Zr on the average grain size of homogenized 7055–xZr–ySc ingots.

The SEM micrographs of the 7055 and 7055–0.25Sc ingots after homogenization are shown in Fig. 6. Micron-sized second phases were present in the two samples. The energy-dispersive X-ray spectroscopy (EDS) spectrum of particle A in Fig. 6(a) revealed that the particle was in the θ -phase. Particle B in Fig. 6(b) contained 61at% Al, 33at% Cu, 1at% Mg, and 5at% Sc, which corresponds to the W-phase.

In the Al–Cu–Sc systems with high Cu contents, Sc atoms diffused into the θ -phase and the process led to the transformation of the θ -phase into the W-phase during homogenization [24–25]. However, the W-phase was not observed in the Al–Cu–Sc alloys when the Cu content was less than 2.5wt% [21–23,27–28]. The Cu content in the 7055 was only 2.24wt%, however, this alloy also contained a high proportion of Zn and Mg atoms, which enhanced the Cu/Al ratio in this system. The extra alloying atoms drove the 7xxx to a nonequilibrium state, which promoted the phase transformation. Thus, the W-phase was formed in the Sc-containing 7055 ingots during homogenization.

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Fig. 5. TEM BF images of homogenized (a) 7055 and (b) 7055–0.25Sc ingots. The arrow in the insert of (b) denotes the diffraction spot of the Al₃(Sc,Zr) phase.



Fig. 6. SEM images of homogenized ingots: (a) 7055; (b) 7055–0.25Sc.

Jia *et al.* [24] reported that all Sc atoms could be dissolved into the W-phase in Al–4.1Cu–0.063Sc alloys. However, Gazizov *et al.* [25] found that the W-phase consumed 50% of the overall Sc atoms in an Al–5.6Cu–0.17Sc–0.12Zr alloy after homogenization. In this study, highly dense precipitated Al₃(Sc,Zr) particles were observed in the homogenized 7055–0.25Sc ingot, indicating that only a small number of Sc atoms were consumed by the W-phase in the 7055–0.25Sc ingot during homogenization.

Fig. 7 shows that the Vickers hardness of the rolled plates varied with the aging time after the plates were treated with a solid-solution. The peak aging time of the 7055-xZr-ySc rolled plates ranged from 24 h to 26 h at $120^{\circ}C$, and the hardness of the 7055-xZr-ySc plates was higher than that of the 7055 plate at all aging time.

Fig. 8 shows OM images of the 7055 and 7055–*x*Zr–*y*Sc after the rolling and T6 treatment. Partial recrystallization occurred in the 7055 specimen, as shown in Fig. 8(a), and

the fibrous grain structure generated by rolling deformation were well preserved in the 7055-xZr-ySc plates after the T6 treatment (Figs. 8(b)-8(f)).



Fig. 7. Variation of Vickers hardness with aging time at 120°C for 7055–xZr–ySc alloys after rolling and solid–solution treatment.



Fig. 8. OM images of the alloys after rolling and T6 treatment: (a) 7055; (b) 7055–0.28c; (c) 7055–0.258c; (d) 7055–0.14Zr–0.158c; (e) 7055–0.14Zr–0.28c; (f) 7055–0.24Zr–0.158c.

Fig. 9 shows the effect of the total mass fraction of Sc and Zr on the short dimension size of the elongated grains of the 7055–xZr–ySc rolled plates after the T6 treatment. The grain size of the samples decreased initially as the total mass fraction of Sc and Zr increased. However, when the total mass fraction of Sc and Zr was greater than 0.45wt%, the grain size did not decrease further and maintained a constant value of approximately 8 µm.

Fig. 10 shows the TEM images of the 7055 and 7055–*x*Zr–*y*Sc rolled plates after the T6 treatment. Highly dense η' precipitates and Al₃Zr dispersoids identified by SAED (inset in Fig. 10(a)) were observed in the BF image of the T6-treated 7055 plate. Highly dense η' precipitates and Al₃(Sc,Zr) particles were also observed in the T6-treated 7055–*x*Zr–*y*Sc plates, in which some of the Al₃(Sc,Zr) particles were present in the GBs and dislocations (Figs. 10(b)

and 10(c)). The size and density of the Al₃(Sc,Zr) particles in the 7055-xZr-ySc plates after the T6 treatment



Fig. 9. Effect of Sc and Zr content on the short dimension size of samples after rolling and T6 treatment.

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Fig. 10. TEM BF images of rolled plates: (a) 7055; (b) 7055–0.25Sc; (c) 7055–0.24Zr–0.15Sc, and DF images of alloys after rolling and T6 treatment: (d) 7055–0.25Sc; (e) 7055–0.14Zr–0.15Sc; (f) 7055–0.24Zr–0.15Sc. The arrows in the inserts of (a) and (b) denote the diffraction spot of the η' phase.

were evaluated by TEM dark-field (DF) imaging. The reflection of the Al₃(Sc,Zr) phase was used during imaging. Highly dense spherical Al₃(Sc,Zr) particles with sizes of 6–18 nm were observed in all of the T6-treated 7055–xZr–ySc plates (Figs. 10(d)–10(f)).

Fig. 11 shows the size distributions of the Al₃(Sc,Zr)

phase in the T6-treated 7055-xZr-ySc rolled plates. The particle size distribution of 11–13 nm were dominant in the three samples, and the amount of Al₃(Sc,Zr) particles with size larger than 13 nm increased with increasing total mass fraction of Sc and Zr in the 7055-xZr-ySc plates. 1566



Fig. 11. Size distributions of the Al₃(Sc,Zr) phase in 7055–*x*Zr–*y*Sc alloys after rolling and T6 treatment.

Fig. 12 shows the STEM high-angle annular dark-field (HAADF) images and the corresponding EDS maps of the T6-treated 7055 and 7055–0.25Sc rolled plates. The T6-treated 7055 plate contained θ -phase (Fig. 12(a)), and

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the EDS results showed that the large particles in the STEM-HAADF image of the T6-treated 7055–0.25Sc plates were in the θ - and W-phases (Fig. 12(b)).

Fig. 13 shows SEM images of the 7055–0.24Zr–0.15Sc rolled plates with different region after the T6 treatment. Micron-sized second phases were observed in this sample. The EDS results revealed that the A particles in region 1 (Fig. 13(a)) contained 64at% Al, 32at% Cu, and 4at% Sc, which corresponds to the W-phase. The B particles in region 2 (Fig. 13(b)) contained 75at% Al, 21at% Zr, and 4at% Sc. This composition and particle size correspond to the primary Al₃(Sc,Zr) phase.

The recrystallization of 7055 rolled plates during the T6 treatment was substantially influenced by the Sc and Zr addition. The Al₃(Sc,Zr) particles located at the GBs strongly pinned the GBs of the 7055–*x*Zr–*y*Sc plates during solution treatment, thereby inhibiting recrystallization and extensive grain growth (Figs. 10(b) and 10(c)).



Fig. 12. STEM-HAADF images and corresponding EDS maps of (a) 7055 and (b) 7055-0.25Sc alloys after rolling and T6 treatment.



Fig. 13. SEM images of the 7055–0.24Zr–0.15Sc alloy after rolling and T6 treatment: (a) region 1; (b) region 2.

The high total mass fraction of Sc and Zr coarsened the precipitated Al₃(Sc,Zr) phase (Fig. 11), and the W-phase (Fig. 12(b)) and primary Al₃(Sc,Zr) phase (Fig. 13) consumed a substantial amount of Sc and Zr atoms and thereby inhibited the increase in the density of the precipitated Al₃(Sc,Zr) phase. Thus, the excessive addition of Sc and Zr (total mass fraction of them larger than 0.45wt%) did not result in further grain refinement in the T6-treated 7055–*x*Zr–*y*Sc plates; the smallest average grain size in the 7055–*x*Zr–*y*Sc plates was approximately 8 μ m when the total mass fraction of Sc and Zr was 0.45wt% (Fig. 9).

Table 3 shows the tensile properties of the T6-treated 7055 and 7055–*x*Zr–*y*Sc rolled plates. The addition of Sc improved the strength and ductility of the 7055 plate. The 7055–0.25Sc plate exhibited a higher ultimate tensile strength (UTS) and a greater elongation (EL) than the 7055–0.2Sc plate. The yield strength (YS) of the 7055 rolled plate was enhanced after the addition of Sc and Zr. However, only the 7055–0.14Zr–0.15Sc rolled plate exhibited superior mechanical properties than those of the 7055 rolled plate. Its YS, UTS, and EL reached 602 MPa, 657 MPa, and 13.8%, respectively. The addition of a large amount of Sc and Zr to the 7055 alloy deteriorated its UTS and EL.

 Table 3. Tensile properties of 7055–xZr–ySc alloys after rolling and T6 treatment

7055–xZr–ySc	Total mass fraction of Sc and Zr / wt%	YS / MPa	UTS / MPa	EL/%
7055	0.16	577 ± 2	654 ± 2	12.8 ± 0.4
7055–0.2Sc	0.36	609 ± 2	649 ± 3	13 ± 0.5
7055–0.25Sc	0.41	600 ± 2	679 ± 2	14.3 ± 0.5
7055-0.14Zr-0.15Sc	0.45	602 ± 3	657 ± 4	13.7 ± 0.8
7055–0.14Zr–0.2Sc	0.5	593 ± 3	635 ± 5	10.3 ± 0.6
7055-0.24Zr-0.15Sc	0.55	580 ± 2	616 ± 3	7.3 ± 0.3

Fig. 14 shows the effect of the total mass fraction of Sc and Zr on the strength and ductility of the 7055-xZr-ySc rolled plates in the T6 state. The YS, UTS, and EL of the 7055-xZr-ySc plates increased initially with increasing total mass fraction of Sc and Zr, and then decreased. The samples with 0.41wt% (7055–0.25Sc plate) and 0.45wt% total mass fraction of Sc and Zr (7055–0.14Zr–0.15Sc plate) exhibited better mechanical properties than the 7055 plate.

The metastable η' phase was the main strengthening phase in the T6-treated 7055 and 7055–*x*Zr–*y*Sc plates. All of the T6-treated 7055–*x*Zr–*y*Sc rolled plates exhibited a higher YS than the T6-treated 7055 rolled plate, primarily

because the enhanced GBs and precipitation strengthening resulted from the fine grains (Figs. 8 and 9) and the increased density of the Al₃(Sc,Zr) particles (Fig. 10).



Fig. 14. Effect of Sc and Zr content on the tensile properties of 7055–*x*Zr–*y*Sc alloys after rolling and T6 treatment.

The strengthening effect of the precipitated $Al_3(Sc,Zr)$ phase on the 7055–*x*Zr–-*y*Sc plates decreased because of coarsening (Fig. 11) with increasing total mass fraction of Sc and Zr. When the total mass fraction of Sc and Zr exceeded 0.45wt%, the grain size maintained a constant value (Fig. 9), and the GBs strengthening effect stopped increasing with increasing total mass fraction of Sc and Zr. Thus, the net effect of high GBs and precipitation strengthening due to the Al₃(Sc,Zr) precipitated phases considerably increased the strength of the 7055–0.25Sc plate.

Notably, the strength of the 7055–0.25Sc plate was only slightly higher than that of the 7055 plate because of the following factors: first, the precipitated Al₃(Sc,Zr) particles were not the main strengthening phase in the 7xxx Al alloys, and the enhancement of precipitation strengthening was limited by the added Sc and Zr. Second, the GBs strengthening effect was not obvious in the Al alloys because of their low Hall-Petch parameters. Third, the W-phase deteriorated the mechanical properties of the high Cu content Al alloys.

After the T6 treatment, the grain sizes of the 7055-xZr-ySc rolled plates were much smaller than those of the 7055 rolled plate (Figs. 8 and 9), and the fine grains in the structure redistributed the stress, thereby preventing strain localization during tension and promoting high elongation before failure. Thus, minor Sc and Zr addition improved the ductility of the 7055 plate (Fig. 14).

The ductility of the 7055-xZr-ySc plates deteriorated when the total mass fraction of Sc and Zr was greater than 0.45wt%. The amount of the micron-sized primary coarse

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Al₃(Sc,Zr) phase was extremely small in the T6-treated 7055–xZr-ySc plates (Fig. 13(b)) because of the low solubility of Zr and Sc in the Al matrix. Thus, the primary coarse Al₃(Sc,Zr) phase led to plastic instability and decreased ductility of the 7055–xZr-ySc plates. In the 7055–xZr-ySc plates with high total mass fraction of Sc and Zr, the dislocation–pinning effect of the precipitated Al₃(Sc,Zr) particles (Fig. 10) weakened because of their large sizes. Thus, the dislocation–accumulation capacities and work-hardening rates of Al alloys during tension decreased [33]. Consequently, the EL of the 7055–xZr-ySc plates decreased as well (Fig. 14).

4. Conclusions

The effect of Sc and Zr microalloying on the microstructure and mechanical properties of a 7xxx Al alloy with a high Cu content was investigated. The following conclusions were obtained:

(1) The addition of Sc and Zr to the 7055 alloy led to the formation of refined and equiaxed grains in the cast ingots. In the homogenized and T6-treated samples, Sc and Zr addition resulted in considerable grain refinement because of the effect of GBs pinning by the precipitated $Al_3(Sc,Zr)$ phase. The Sc atoms diffused into the θ -phase and promoted the formation of the W-phase in the 7xxx Al alloy with a high Cu content.

(2) The peak aging time of the 7055 and 7055–xZr–ySc rolled plates ranged from 24 h to 26 h at 120°C, and the hardness of the 7055–xZr–ySc alloys was greater than that of the 7055 alloy.

(3) Minor additions of Sc and Zr improved the mechanical properties of the T6-treated 7055 plate because of the refined grains and increased density of the fine Al₃(Sc,Zr) phase. However, the W-phase and primary Al₃(Sc,Zr) phase, as well as the insufficient amount of refined grains, deteriorated the mechanical properties of the T6-treated 7055–*x*Zr–*y*Sc rolled plates when the total mass fraction of Sc and Zr exceeded 0.45wt%. The 7055–0.25Sc rolled plate exhibited the best mechanical properties among the prepared alloys.

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