



Effect of aging treatment on evolution of S' phase in rapid cold punched Al–Cu–Mg alloy

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Abstract: High-resolution transmission electron microscopy (TEM), X-ray diffractometer (XRD), and hardness test were used to study the evolution of long plate-shaped S' phase in the spray-formed fine-grained Al–Cu–Mg alloy during aging after rapid cold punching deformation. Results show that the long plate-shaped S' phase in the extruded Al–Cu–Mg alloy undergoes evident distortion, brittle failure, separation and redissolution, during rapid cold punching deformation, leading to the transformation of long plate-shaped S' phase into short rod or even redissolution and disappearance, causing the matrix to become a supersaturated solid solution. After the aging treatment, the reprecipitation of the phases occurs, and these aging phases are mainly long plate-shaped and granular. The incompletely dissolved S' phase acts as nucleation core, promoting uphill diffusion of the surrounding solute atoms. The S' phase gradually grows with increasing the aging time. The completely dissolved S' phase forms the incoherent equilibrium phase with the matrix to reduce its free energy. After rapid cold punching, the aging response of the deformed Al–Cu–Mg alloy is accelerated, and the hardness of the alloy is substantially increased.

Key words: aging treatment; Al–Cu–Mg alloy; rapid cold punching; dissolution; reprecipitation

1 Introduction

Grain refining is the main method for improving strength and toughness of metal. Severe plastic deformation as an effective method to refine grain has always been a hot research point in the field of materials [1–9]. Researchers have found that in addition to refining grain, severe plastic deformation induces distortion, brittle failure and even redissolution of second-phase in the alloy, which transform the matrix into a supersaturated solid solution [10–15]. NOURBAKHS and NUTTING [16] have found that the acicular θ' phases are completely broken into small spherical particles as rolling reduction exceeds 60% in the Al–4%Cu alloy, and high density and uniform

dislocations are also observed. MURAYMA et al [17] have discovered that the matrix of equal-channel angular-pressed Al–Cu alloy transforms to supersaturated solid solution after precipitation redissolution. During aging, the precipitation of equilibrium phase θ directly differs from the regular order of aging precipitation. FAN et al [18] have reported that rapid cold punching deformation causes the breakage of thick long strip-shaped S' phase and needle-like θ' phase in the as-extruded alloy and promotes the dissolution of the nano-precipitate phase. The high-distortion free energy of the matrix promotes the precipitation of the equilibrium θ phase after redissolution and reprecipitation. ZHANG et al [19] have observed the redissolution of precipitation in the multi-directional forged Al–Cu alloy, and have found that

the strain-induced precipitation in supersaturated solid solution is greatly accelerated during re-aging. The precipitation sequence is related to heating temperature, deformation degree, and grain size after deformation. XU et al [20] have investigated the redissolution and reprecipitation behavior of θ' phase in Al–Cu alloy via equal-diameter extrusion deformation at a normal temperature. They have found that when the heating temperature is sufficiently high to counteract the high internal stress resulting from severe plastic deformation, the precipitation sequence is from metastable phase to stable phase. When the heating temperature is insufficiently high to counteract the high internal stress and the grains are ultra-refined, the precipitation sequence is that equilibrium phases are formed directly instead of metastable phases. In addition, the supersaturated solid solutions are formed after redissolution of precipitated phases induced by severe deformation. On this basis, the aging treatment is expected to obtain the precipitated phase with significantly dispersion-strengthened effect to achieve the effective unity of fine grain strengthening, deformation strengthening, and precipitation hardening.

In-depth research on the reprecipitation behavior in the aging process after precipitation redissolution during severe deformation has great significance for the study about the strengthening mechanism of the Al–Cu alloy. However, the existing literature focuses on the influence of plastic deformation on the characteristics of θ phase in the Al–Cu alloy and the redissolution mechanism. The influence of aging treatment on the evolution of S' phase in severe plastic deformation of Al–Cu–Mg alloy is rarely discussed. To explore this

phenomenon, the aging reprecipitation behavior after dissolution of the precipitated phase during rapid cold punching deformation was studied with rapid solidified fine-grained Al–Cu–Mg alloy billet by spray forming as basis.

2 Experimental

A rapidly solidified fine-grained Al–Cu–Mg alloy cylindrical ingot was prepared on a self-developed SD380 large-scale injection molding apparatus. Table 1 shows the chemical composition of the alloy. The cylindrical ingot was extruded into a $\phi 30$ mm round bar on a 1250 T extruder at 450 °C with an extrusion ratio of 15:1. The round bar was cut into a small cylinder of $\phi 30$ mm $\times$ 20 mm. The extruded sample was placed in a self-designed punching mold for 1–4 passes of rapid cold punching at 25 °C. The sketch maps of rapid cold punching are illustrated in Fig. 1. The samples after various passes of rapid cold punching were artificially aged at 181 °C for 1–20 h and air cooled. The process parameters of rapid cold punching are listed in Table 2.

The samples were obtained on the central wall of the rapid cold forming cylinder for micro-structure observation. The morphology, size, and distribution of the nano-precipitated phase of the sample were analyzed through a JEM–F200 transmission electron microscope (TEM). The

Table 1 Chemical composition of Al–Cu–Mg alloy (wt.%)

Cu	Mg	Mn	Si	Fe	Al
4.51	1.46	0.56	<0.05	<0.05	Bal.

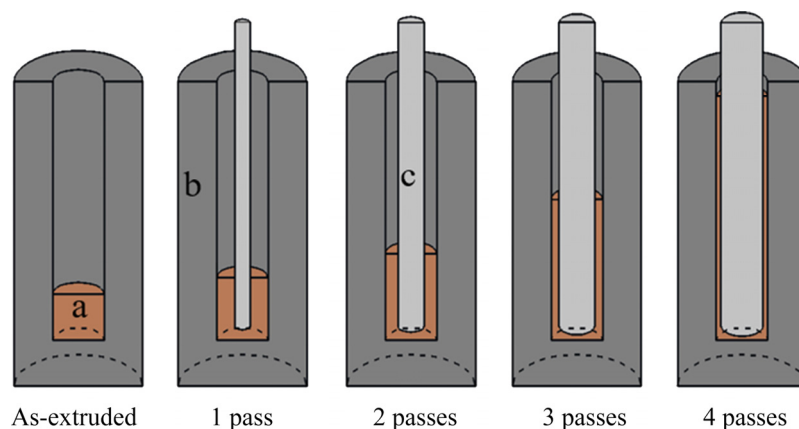


Fig. 1 Schematic of rapid cold punching (a—Sample; b—Drawing die; c—Punch)

Table 2 Process parameters of rapid cold punching

Punch pass	Diameter/mm	Velocity/(mm·s ⁻¹)
1	10	30
2	14	25
3	20	20
4	27	15

sample for TEM observation was mechanically pre-thinned to approximately 80 μm , and then twin-jet electro-polished with a solution of nitric acid and methanol (volume ratio of 1:3) at a temperature lower than $-25\text{ }^{\circ}\text{C}$. The electron microscopy parameters of the high-angle annular dark field scanning transmission electron microscope were as follows: acceleration voltage of 200 keV, electron beam half-convergence angle of 10 mrad, high-angle annular probe for collecting the inner half angle of 36 mrad, and a beam spot diameter of 0.20 nm. The phase identification was analyzed by a D/max 2500 18 kW target X-ray diffractometer. Vickers microhardness was measured using a pyramid diamond indenter to track the evolution of aging hardening with a load of 100 g and a holding time of 10 s. At least 15 points were measured for each sample and the measurement position was the middle area of the outer surface of the cylinder wall.

3 Results

3.1 Aging hardening curves

Figure 2 shows the hardening curves of extruded Al–Cu–Mg alloys after various rapid cold punching passes subjected to aging treatment at $181\text{ }^{\circ}\text{C}$. During aging, the extruded samples with rapid cold punching show higher hardness than those without rapid cold punching. As the number of rapid cold punching pass increases, the peak hardness of aging samples substantially increases, and the aging response is greatly accelerated. It can be seen that the aging sample with 4 rapid cold punching passes exhibits the largest peak hardness (HV 193) and the shortest aging response time (2 h). Thus, rapid cold punching deformation not only improves the hardness of the Al–Cu–Mg alloy, but also shortens the time to reach peak hardness.

3.2 XRD patterns

Figure 3 depicts the XRD patterns of the

peak-aged Al–Cu–Mg alloy samples after various rapid cold punching passes. The diffraction peaks show that the main precipitates are $\alpha(\text{Al})$ phase and S' phase (Al_2CuMg), and the diffraction peak intensity of S' phase tends to increase with increasing the number of rapid cold punching passes.

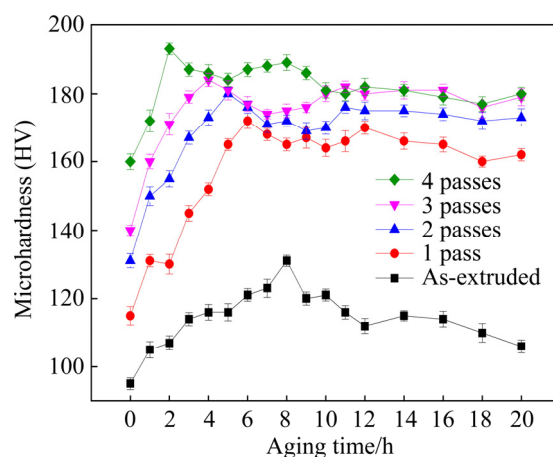


Fig. 2 Evolution of microhardness of extruded Al–Cu–Mg alloy subjected to aging treatment after various passes of rapid cold punching

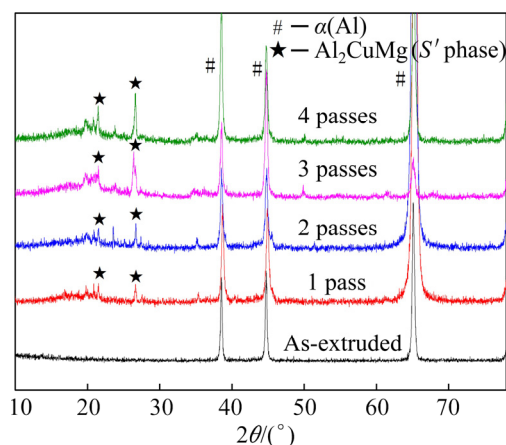


Fig. 3 XRD patterns of peak-aged Al–Cu–Mg alloy samples after various rapid cold punching passes

3.3 TEM observation

Figure 4 presents the TEM bright field images of the peak-aged Al–Cu–Mg alloy samples after various rapid cold punching passes. The precipitated phase in the extruded sample is mainly long plate-shaped S' phase. With increasing the number of rapid cold punching passes, the size of the S' phases shortens, the density increases and the distribution becomes more uniform in the peak-aged samples. The elongated S' phases in the as-extruded alloy are large and regularly distributed

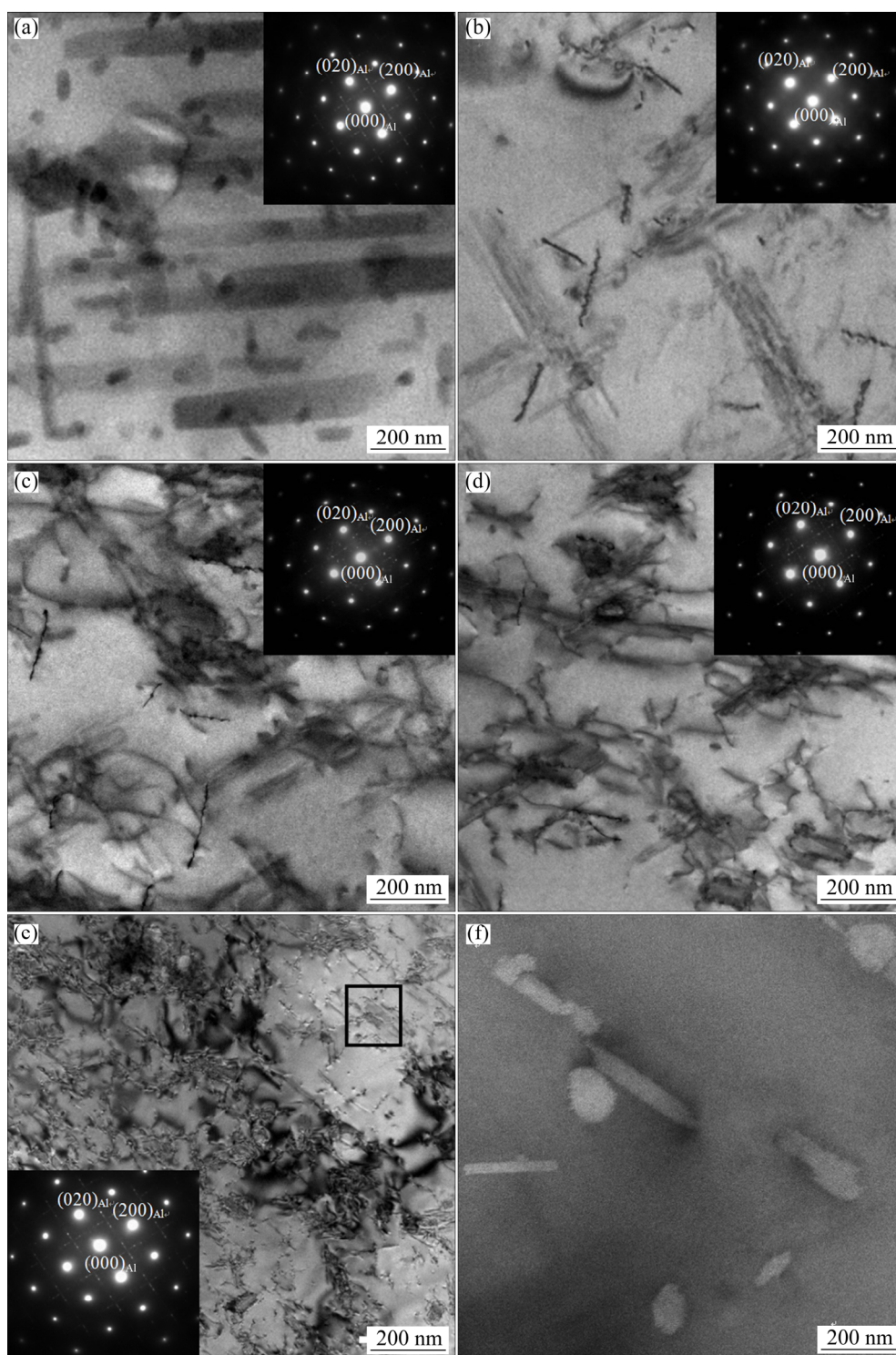


Fig. 4 TEM bright field images (a–e) and HAADF–STEM image (f) of peak-aged samples of S' precipitated phase undergoing extrusion (a) and various passes of rapid cold punching deformation: (b) 1 pass; (c) 2 passes; (d) 3 passes; (e) 4 passes; (f) HAADF–STEM image of square area in Fig. 4(e) (The insets in (a–e) are SAED patterns)

along the extrusion direction. After one and two passes of rapid cold punching, the size of long plate S' phases in peak-aged samples evidently shortens, the dislocation density increases considerably, and is mainly distributed surrounding S' phases as

shown in Figs. 4(b) and (c), respectively. The S' phases in peak-aged samples are refined, and the number is further increased up to 3 passes, as shown in Fig. 4(d). After 4 passes, the size of the S' phases is significantly refined, and the distribution

is more uniform (see Fig. 4(e)). Figure 4(f) shows the HAADF–STEM image of the square area in Fig. 4(e), which reveals that the morphology of the S' phases is mainly long plate and granular. The insets in Figs. 4(a–e) are the electron diffraction patterns along the $\langle 001 \rangle_{\text{Al}}$ crystal ribbon axis. The orientation relationship between the S' phase and the aluminum matrix is $[001]_{S'}/[001]_{\text{Al}}$.

4 Discussion

4.1 Evolution mechanism of S' phase during rapid cold punching

The main strengthening phase in extruded Al–Cu–Mg alloy is the S' (Al_2CuMg) phase, which is regularly distributed in the aluminum matrix. The S' phase is flat with the aluminum matrix (Fig. 5(a)). After rapid cold punching deformation, the long plate-shaped S' phase is distorted under strong shear deformation force, causing the high tendency of the

solute atoms located in the region of the S' phase with high distortion energy to be redissolved into the aluminum matrix (Fig. 5(b)).

As deformation continues, the distortion of the long plate-shaped S' phase further intensifies, and a significant “resolved step” appears on the S' phase. As the “resolved step” continues to increase (Fig. 5(c)), the brittle interfaces increase evidently, which then substantially increase the contact surface between the precipitated phase and the aluminum matrix. This situation promotes the dissolution on the S' phase, leading to a considerable reduction in the size of the S' phase in this region and forming necking. The necking further increases the interface distortion energy between the S' phase and the aluminum matrix, accelerating the redissolution at the necked position until it disappears. The disappearance of the necking separates the two parts of the precipitated phase, forming many short rod-like precipitated

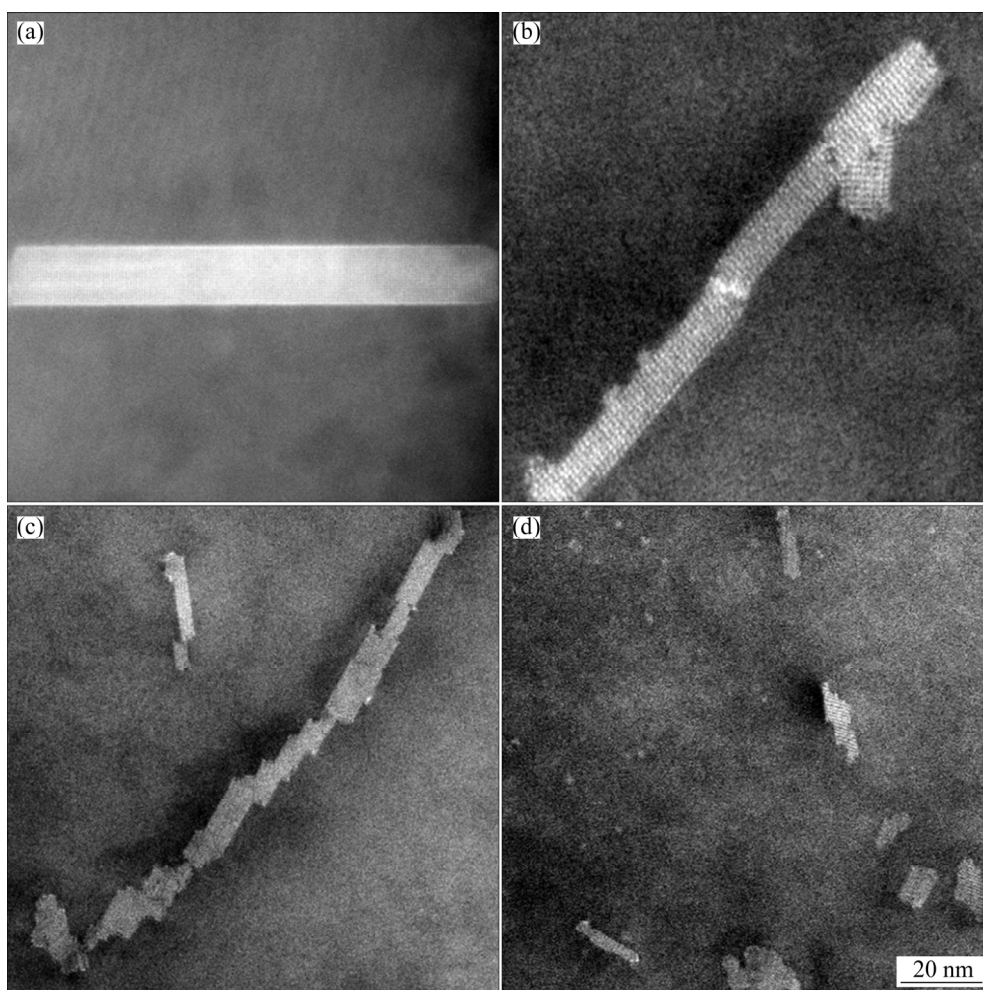


Fig. 5 HAADF–STEM images of S' precipitated phase undergoing extrusion (a), 2 passes (b), 3 passes (c), and 4 passes (d) of rapid cold punching deformation, viewed along $[001]_{\text{Al}}$ direction

phases that are distributed in the aluminum matrix (see Fig. 5(d)). The contact surface between the precipitated phase and the aluminum matrix is evidently increased, and the interfacial distortion energy between the precipitated phase and the aluminum matrix is improved. The free energy of the S' phase becomes higher than that of the matrix. The energy balance between the pre-precipitated phase and the matrix is broken, creating conditions for the solute atoms to redissolve into the matrix. In addition, the average grain size of the experimental alloys fabricated by the spray-forming rapid solidification technology is roughly $5\ \mu\text{m}$ with uniform structure.

During rapid cold punching deformation, the grains are further refined to nano scale. Thus, the grain boundary area increases, which provides more atomic diffusion channels for the redissolution of the precipitated phase and greatly promotes the redissolution of S' phase.

4.2 Dissolution mechanism of S' phases during rapid cold punching

Based on the contact dislocation mechanisms of the phase dissolution after the metal deformation [21], severe plastic deformation creates massive dislocations that are hindered by the precipitated phase during the movement. This situation promotes dislocation pileup and forms Cottrell atmosphere that absorbs numerous heterogeneous solute atoms. Such an atmosphere provides a channel for the redissolution of the precipitated phase. The model is shown in Fig. 6.

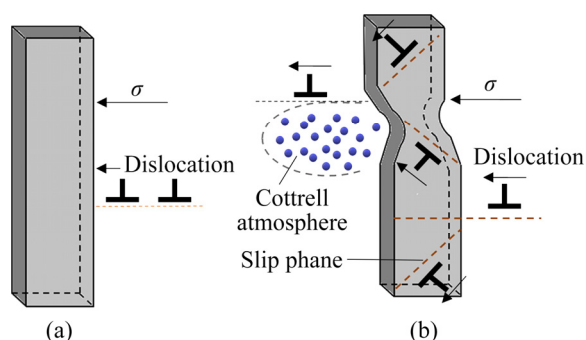


Fig. 6 Mechanism of dissolution of second phases upon contact interaction with dislocations: (a) Initial state, dislocations moving to dissolving phase under action of shear stress σ ; (b) Mechanism of phase shearing upon dissolution (shaded signs are moving dislocations, dashed lines are slip planes, and points around the dislocation symbol are Cottrell atmospheres of the solute atoms carried from the second phase)

During rapid cold punching deformation, the twist and brittle fracture of long plate-shaped S' phase evidently increases the contact surface between the precipitated phase and the aluminum matrix. The interfacial distortion energy between the two is improved as well, providing energy for the migration of Cu and Mg atoms. As the number of passes of rapid cold punching increases, dislocations also increase considerably. Those dislocations are hindered and piled up by the S' phase during the movement, leading to more and more Cottrell atmospheres of the solute atoms carried from the second phase. At last, massive Cu and Mg atoms are redissolved to Al matrix.

Moreover, upon the plastic deformation, dislocations are easily generated by a great number of Frank–Read dislocation sources, which continuously generate new dislocations under strain [22]. The Cottrell atmospheres are generated around dislocations and locally eliminate the solute atoms. This process, in turn, disrupts the equilibrium distribution of the solute atoms in the volume of the matrix. This results in the generation of diffusion fluxes directed toward the dislocation atmospheres, which attempt to equalize the solute atom concentration in the entire volume of the solid solution. However, the equilibrium between the precipitated phase and the matrix is broken, because the solute atom concentration balance between the precipitated phase and the matrix is disrupted. Thus, a thermodynamic stimulus arises for the dissolution of the phase back into the matrix [23]. The model is shown in Fig. 7. Figure 5(c) shows that the S' phase

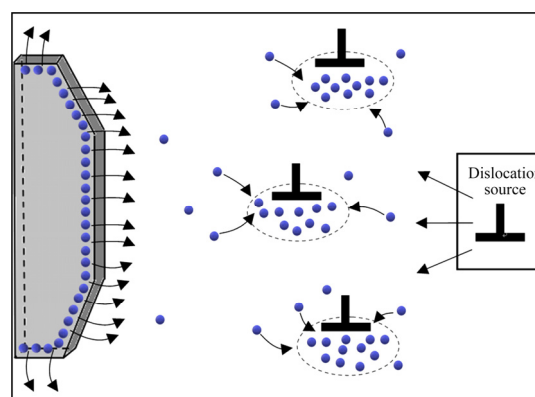


Fig. 7 Schematic diagram showing phase-dissolution mechanism upon noncontact interaction with dislocation (circles outline Cottrell atmospheres, dots represent solute atoms, and arrows in the phase show the escape directions of the solute atoms from the phase upon dissolution)

dissolution begins at the corners, indicating that the Cu and Mg atoms are dissolved from the corners into the aluminum matrix. This phenomenon proves that the S' phase dissolution during rapid cold punching is consistent with the non-contact mechanism of the dislocation-induced phase dissolution [21].

4.3 Precipitation behavior of S' phase during artificial aging after rapid cold punching deformation

Under the condition of rapid cold punching deformation, the long plate-shaped S' phase in the extruded Al–Cu–Mg alloy undergoes intense distortion, brittle fracture, redissolution, necking, and separation. Consequently, the long strips of S' phases transform into short rods or even redissolve and disappear. The experimental results show that after dissolution of the S' phase induced by rapid cold punching, reprecipitation occurs during aging, and the morphology of the aged precipitate phase is mainly long plate-shaped and granular (see Fig. 4(f)).

Given that the diffusion in solids relies on thermally activated motion of atoms, atoms can be activated for the migration only at high temperatures [24]. Rapid cold punching is carried out under low temperature, high strain, and high strain rate, so atom diffusion is weak, diffusion distance is short, and most of the Cu and Mg atoms remain clustered around S' phase before redissolution. The size of the S' phase in the extruded sample is different, so the extent of redissolution during rapid cold punching deformation is also different. When the deformation stops, a small part of the S' phase will not be completely dissolved. During aging, the incompletely dissolved S' phase acts as nucleation core, promoting uphill diffusion of the surrounding solute atoms. As aging time increases, the S' phase will gradually grow. Figure 4(f) shows that the long plate-shaped S' phase having a semi-coherent relationship with the matrix is precipitated during aging, as well as the granular S phase that is incoherent with the matrix. The increase of distortion-free energy caused by the fracture of the long plate-shaped S' phase and the deformation energy stored during rapid cold punching deformation substantially increases the free energy of the system. The aging temperature is insufficient

to eliminate the severe lattice distortions of the matrix after deformation. However, the precipitation of coherent or semi-coherent transition phases cannot be spontaneously performed because the interfacial relationship between these transition phases and the matrix causes further increase of strain energy in the alloy, which results in a high free energy of the system. Therefore, the formation of an equilibrium phase having no coherent relationship with the matrix at the grain boundary is advantageous for reducing the free energy.

In addition, the grains are further refined into nano scale, and lattice defects such as grain boundaries and dislocations increase geometrically during rapid cold punching deformation. Thus, the non-uniform nucleation rate of the S phase also increases geometrically. The solute atoms in the inner matrix quickly diffuse to the heterogeneously nucleated stable phase, causing rapid disappearance of the solute supersaturation required to form the GP region and metastable phase in the crystal. Moreover, the driving force for phase transformation strongly declines. The dislocation density increases substantially in the rapid cold deformation of the experimental alloy. Numerous vacancies are produced in the matrix during delivery and movement of the dislocation. Existence and movement of vacancies create conditions for atomic diffusion. Every pass of a newly rapid cold punching deformation produces numerous vacancies, which promote the diffusion of solute atoms. The amount of S' phases considerably increases, and the size is smaller in the aged samples with rapid cold punching than that without rapid cold punching.

5 Conclusions

(1) After rapid cold punching, the aging response of the deformed Al–Cu–Mg alloy is accelerated, and the hardness of the alloy is substantially increased. Compared with the extruded sample, which reaches the peak hardness of HV 131 after 8 h aging, the peak hardness of the extruded sample with 4 passes of rapid cold punching increases to HV 193 (about 47.3% increment) at aging time of 2 h (shortened by 6 h).

(2) During rapid cold punching deformation, the dissolution mechanism of the long plate-shaped

S' phase in the deformed Al–Cu–Mg alloy mainly includes contact dislocation mechanism and non-contact dislocation-induced mechanism.

(3) After dissolution of the S' phase induced by rapid cold punching, the matrix becomes resaturated. The reprecipitation of the phases occurs, and these aging phases are mainly long plate-shaped and granular. The incompletely dissolved S' phase acts as nucleation core, promoting the uphill diffusion of the surrounding solute atoms. The S' phase gradually grows with increasing the aging time. The completely dissolved S' phase forms the incoherent equilibrium phase with the matrix to reduce the free energy of the matrix.

Acknowledgments

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时效处理对快速冷冲 Al–Cu–Mg 合金 S'相演变规律的影响

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摘 要: 采用高分辨透射电镜(TEM)、X 射线衍射仪(XRD)和硬度测试等手段, 研究快速冷冲后时效过程中喷射成形细晶 Al–Cu–Mg 合金中长片状 S' 相的演变规律。研究表明: 快速冷冲过程中, 挤压态 Al–Cu–Mg 合金中长片状 S' 相发生明显的扭曲、破断、分离和回溶, 导致长片状 S' 相变成短棒状, 甚至完全回溶, 使基体成为过饱和固溶体。经时效处理后发生再析出, 时效析出相形貌以长片状和颗粒状为主。在时效过程中未完全回溶的 S' 相作为形核核心, 使周围溶质原子发生上坡扩散, 随着时效时间的延长, S' 相逐渐长大; 部分 S' 相完全回溶后在时效过程中析出与基体不共格的平衡相, 以降低基体的自由能。挤压态 Al–Cu–Mg 合金经快速冷冲后, 时效响应速度加快, 合金硬度显著增加。

关键词: 时效处理; Al–Cu–Mg 合金; 快速冷冲; 回溶; 再析出

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