



Al-Cu 合金纳米析出相回溶和再析出的研究现状及发展趋势

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摘要: 综述了 Al-Cu 合金在强塑性变形过程中纳米析出相的演变规律, 室温强塑性变形诱导 Al-Cu 合金析出相回溶, 导致基体重新形成过饱和固溶体, 继续对合金进行强塑性变形或时效处理, 基体中析出再析出相, 合金的力学性能显著提高。综合分析析出相回溶的机理、机制及回溶和再析出对 Al-Cu 合金组织和性能的影响, 阐述了 Al-Cu 合金纳米析出相回溶和再析出的研究方向和发展趋势, 以期为制备性能优异的 Al-Cu 合金材料提供理论参考。

关键词: Al-Cu 合金; 纳米析出相; 回溶; 再析出

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0 引言

Al-Cu 合金因具有强度高、加工成形性好及耐热性优良等特点, 已被广泛应用于航空航天及军工领域, 但 Al-Cu 合金的韧性、硬度等性能仍有待改善^[1-3]。强塑性变形技术作为改善铝合金微结构、获得微米甚至纳米级细晶材料的重要手段, 越来越引起国内外学者的关注^[4-9]。室温下强塑性变形可使 Al-Cu 合金中第二相质点发生回溶, 使基体重新成为过饱和固溶体, 继续对合金进行强塑性变形或时效处理, 新的第二相以细小颗粒析出, 从而显著提高合金的力学性能^[10-15]。

为了深入探讨 Al-Cu 合金纳米析出相回溶和再析出行为, 本研究综述了国内外学者对 Al-Cu 合金在强塑性变形过程中纳米析出相回溶和再析出的研究成果, 分析了析出相回溶的机理、机制及回溶和再析出

对 Al-Cu 合金组织和性能的影响, 阐明了 Al-Cu 合金纳米析出相回溶和再析出的研究方向和发展趋势, 旨在为深入研究 Al-Cu 合金的强韧化机制及制定新的强塑性变形工艺提供理论参考。

1 析出相回溶

在室温下 Al-Cu 合金经强塑性变形时, 析出相往往会产生塑性变形、破碎, 甚至回溶到基体中形成过饱和固溶体, 从而对合金的性能产生影响。为了满足航空航天及汽车行业等对高强钢 Al-Cu 合金材料的需求, 关于 Al-Cu 合金回溶扩散过程中, 原子回溶到基体中的途径和方式、回溶机理以及在基体中形核和长大的机制仍需深入探讨。

1.1 强塑性变形诱导析出相回溶现象

强塑性变形通常有等径角挤压 (equal channel angular pressing, ECAP)、多向压缩 (multi-axial

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compression, MAC) 等方法。ECAP 是通过强烈塑性变形使材料的晶粒被剪切细化, 从而获得超细晶或纳米晶材料; 而 MAC 是通过改变压缩过程中的应变轴 $X \rightarrow Y \rightarrow Z \rightarrow X$ 进行多向压缩, 以获得基体的均匀变形^[16]。

国内外学者对 Al-Cu 合金强塑性变形过程中的微结构演化规律进行了大量的研究。S. Nourbakhsh 等^[17]对 Al-4%Cu 合金进行高压下冷轧后发现, 合金的片状 θ' 相发生严重弯曲和破碎。O. N. Senkov 等^[18]最先发现在低温强塑性变形过程中, Al-Fe 合金中 Al_3Fe_4 析出相的回溶现象。M. Murayama^[19]在 Al-Cu 合金的室温下 ECAP 变形中发现, Al-Cu 合金呈针状的 θ' 过渡相在数道次 ECAP 过程中逐步分解成短链状颗粒, 直至回溶进入基体。彭北山等^[20]研究发现, Al-Cu 合金的 θ' 相经 ECAP 挤压 8 道次后, 因自身应变作用, 在合金内部形成了位错、亚晶粒、剪切带和扭转带等缺陷, 并在这些缺陷与基体的界面处发生溶解、断裂、碎化和球化, 直至溶入基体中。

胡楠等^[21]在 MAC 变形下, 分别以含 θ' 相和含 θ'' 相的 Al-Cu 合金试样为研究对象进行观察。含 θ'' 相试样经 MAC 变形 1 道次时, θ'' 相不变形; 变形 4 道次时, 析出相数量减少, 由此说明析出相开始大幅度溶解; 变形 8 道次, 已观察不到析出相。含 θ' 相试样经 MAC 变形 1 道次时, θ' 相明显变形; 随着应变量的增加, 变形 4 道次时, 析出相明显破碎; 当 MAC 变形 8 道次时, 析出相的数量减少, θ' 相部分回溶; 变形至 12 道次时, 几乎不存在可辨别的 θ' 相; 变形 16 道次时, 几乎看不到 θ' 相, 说明析出相基本回溶至基体中。

党鹏等^[16]研究了在 ECAP 和 MAC 两种强塑性变形下, θ' 相变形的演变规律。在相同应变条件下, 就析出相回溶速度而言, MAC 变形明显快于 ECAP 变形。当应变增加到一定程度时, θ' 相与基体失去位向关系, 应变进一步增加, θ' 相发生变形、破碎和回溶。Huang W. J. 等^[22-23]以 Al-Cu 合金的 θ'' 相为研究对象, 探索 θ'' 相在两种强变形方式 (ECAP 和 MAC) 中相似应变下的演变规律。在 MAC 变形中, 应变量为 0.8 时, 组织中 θ'' 相粒子大量溶解; 应变增加到 2.4 后, θ'' 相粒子已经完全回溶到基体中。而在 ECAP 变形中, 应变量为 0.7 时析出相仅仅发生弯曲, 几乎没有溶解; 应变量为 2.8 时还存在少量的

θ'' 相粒子。这说明在应变量相似条件下, θ'' 相粒子在 MAC 过程中回溶速度比 ECAP 快。

1.2 回溶机理

强塑性变形下合金析出相回溶的机理存在不同观点, 大致有以下 4 种。1) M. Murayama^[19]认为, 在强塑性变形过程中, θ' 相被弯曲、破碎, 导致 θ' 相的表面能增加, 从而使亚稳相 θ' 回溶于基体, 形成过饱和固溶体。2) D. Fátay 等^[24]研究结果表明, 经 15 次高压扭转后, X 射线衍射图上未能观察到析出相的衍射斑点, 这是由于析出相被粉碎, 在基体晶界处形成一个薄层, 并非析出相回溶于基体内形成过饱和固溶体。3) H. Hidaka 等^[25]以 Fe-C 合金为研究对象, 球磨后共析钢中的渗碳体全部消失, 位于铁素体中的碳含量较低, 其余的碳都处于铁素体晶界处。4) P. Hewitt 等^[26]根据 Gibbs-Thompson 方程认为, 基体中小于平均直径的细晶周围溶解度较高, 超过基体的平均浓度从而产生回溶。

若按照观点 1), 在强塑性变形条件下晶界表面能增加应导致晶界消失, 但强塑性变形形成超细晶后, 晶界并未消失, 其数量反而增加。P. J. Apps 等^[27]报道析出相回溶后引起基体的晶格常数增大, 由此说明 D. Fátay、H. Hidaka 等提出的观点 2) ~3) 没有普遍意义, H. Hidaka 等只是说明了渗碳体的去处, 但未阐明渗碳体的溶解原理。析出相的宏观溶解度不会因为在加热过程中细小粒子的溶解而产生改变^[28-30], 由此表明观点 4) 也不能说明所有现象。因此以上 4 种观点不能完全阐明在强塑性变形条件下析出相回溶的机理, 机理研究仍需要进一步探索。

1.3 回溶机制

不论变形方式属于哪种类型, 析出相回溶都具有以下特征: 1) 变形量达到一定程度时, 析出相回溶现象才能发生; 2) 析出相回溶的驱动力来源于变形量; 3) 变形量越大析出相回溶的速度越快。在 Al-Cu 铝合金中, 亚稳相 (θ'' 、 θ') 回溶所需的变形量低于平衡相 θ , 即析出相与基体不同程度的共格导致其所需的变形量不同^[31-39]。析出相回溶的速度受变形速率影响, Al-Cu 合金析出相回溶在高变形速率下所需的变形量比在低变形速率下小。

彭北山等^[40]认为 θ' 相是先溶解导致破碎球化而不是先破碎球化再溶解。其主要演变过程为: 在剪切应力作用下发生弯曲变形, 从而形成了大量亚结构; 高能

态的亚结构界面为 θ' 相回溶提供了扩散通道, θ' 相沿亚结构界面能快速溶解,而不是粒子破碎球化至临界形核尺寸之下才溶解。

析出相回溶的主要机制为:一是室温下应变引起的界面反应型回溶机制和界面弯曲效应机制;二是温度起主要作用的扩散控制型回溶机制^[41]。在强塑性变形下,一方面基体与析出相界面产生大量位错,界面与位错相互作用;另一方面使析出相变形、破碎,且界面弯曲效应增强,这促进了析出相的回溶^[42-45]。

2 再析出析出相

Al-Cu合金在强塑性变形过程中,析出相发生回溶,导致基体重新形成过饱和固溶体,继续对合金进行强塑性变形或人工时效处理,铝基体中再析出析出相,合金的力学性能得到显著提高。

M. Murayama^[19]认为由强塑性变形引起Al-Cu合金中的过饱和固溶体再析出析出相的析出顺序与常规态不同,平衡相直接在晶界上析出,而过渡相的析出被抑制。但V. M. Segal等^[46]则认为,强塑性变形并未改变再析出顺序,仅仅加快再析出析出相。Xu X. C.等^[47]研究强塑性变形诱导Al-Cu合金析出相回溶时发现,过饱和固溶体再析出存在两种方式:一是强塑性变形再析出;二是人工时效再析出。

2.1 强塑性变形再析出

张孜昭等^[48]研究表明,Al-Cu合金的过饱和固溶体经强塑性变形会再析出,这是因为强塑性变形使Al-Cu合金内产生大量位错和晶格畸变,从而使合金内畸变能剧增,所以发生再析出;继续变形时过程中亚稳相 θ'' 和 θ' 的析出被抑制,直接在晶界处析出平衡相 θ 。已有研究表明^[49-54],强塑性形变会导致Al-Cu合金析出相回溶形成过饱和固溶体,同时又会促进过饱和固溶体脱溶,形成 θ 相。在继续变形过程中,当 θ 相自由能高于基体自由能时, θ 相会再次回溶于基体^[55-66]。

Fan C. H.等^[67]研究了快速冷冲强变形过程中喷射成形细晶Al-Cu-Mg合金纳米析出相回溶及再析出行为。不同道次快速冷冲后Al-Cu-Mg合金的XRD谱如图1所示。由图1可见,随着快速冷冲道次的增加,合金基体中析出相的峰值大小呈现先减小再增大的变化趋势。经2道次快速冷冲强变形后Al-Cu-Mg合金中 S' 相发生明显的破碎和分离(见图2a~b),

θ' 相发生弯曲、折断和分离(见图2c~d)。经3道次快速冷冲强变形后挤压态合金中的长条状 S' 相和针状 θ' 相基本回溶,形成过饱和固溶体。4道次快速冷冲Al-Cu-Mg合金析出相HAADF-STEM图和EDS图如图3所示。由图3可见,挤压态Al-Cu-Mg合金经4道次快速冷冲强变形后发生再析出行为,合金基体中再析出相主要为颗粒状平衡相 θ ,很少观察到平衡相 S 。

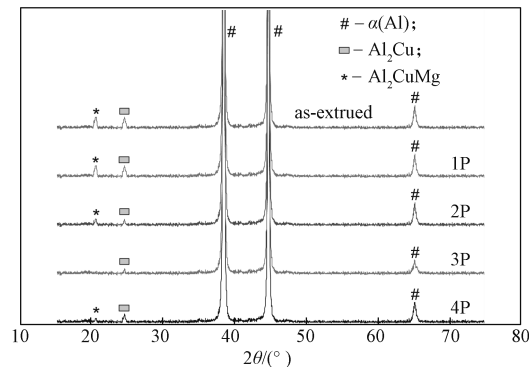


图1 不同道次快速冷冲后Al-Cu-Mg合金XRD谱
Fig. 1 XRD patterns of Al-Cu-Mg alloy samples undergoing various passes of rapid cold stamping process

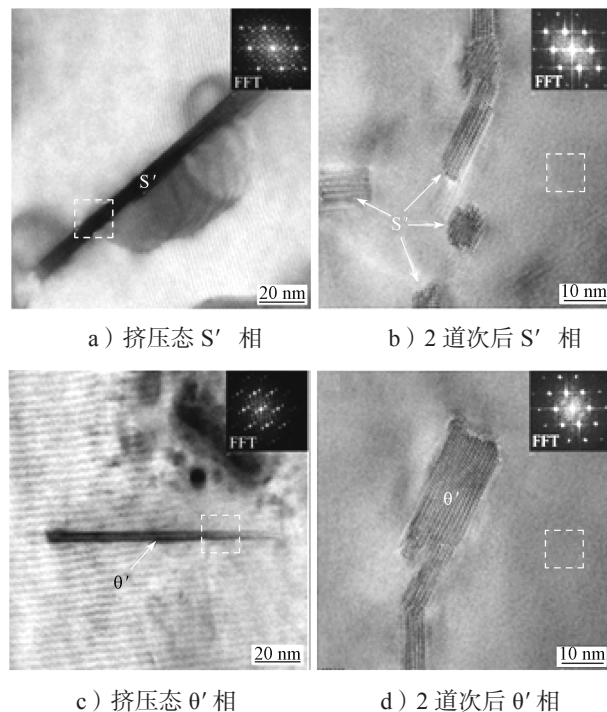


图2 挤压态和经2道次快速冷冲变形后 S' 相、 θ' 相TEM及FFT图

Fig. 2 TEM and FFT images of S' and θ' precipitated phase undergoing extrusion and 2-pass of rapid cold stamping deformation

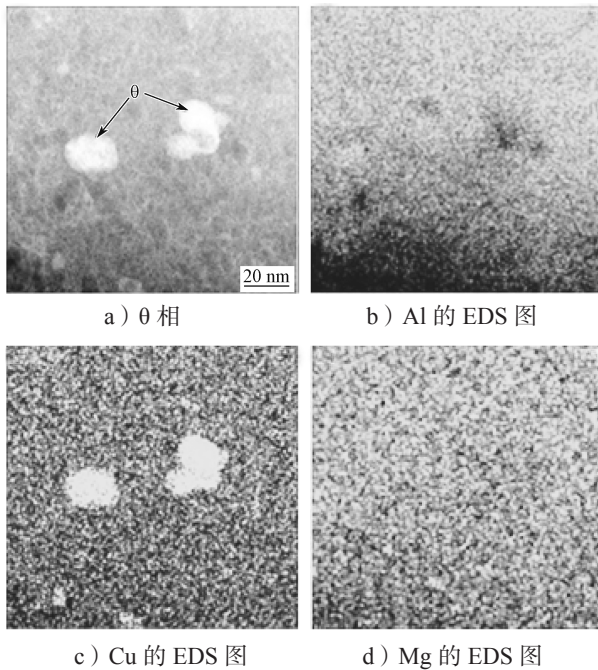


图 3 4 道次快速冷冲后 Al-Cu-Mg 合金析出相 HAADF-STEM 图及 EDS 图

Fig. 3 HAADF-STEM image and EDS maps of precipitated phase in Al-Cu-Mg alloy undergoing 4-pass

2.2 人工时效再析出

在人工时效与强塑性变形过程中, Al-Cu 合金析出相的再析出行为不同, 因为人工时效再析出的驱动力是温度, 强塑性变形再析出的驱动力是形变能^[68-73]。当加热温度高至足够消除基体的晶格畸变时, 再析出顺序与常规态相同^[74-75]; 当加热温度不足以消除基体的晶格畸变时, 再析出顺序发生变化^[76-77]。

张孜昭等^[78] 研究表明, 当试样变形程度小于 22 道次时, 200 °C 的时效温度基本可以消除基体的晶格畸变, 从而使试样析出顺序未发生改变, 只是加快再析出析出相的速度; 当试样变形 30 道次时, 200 °C 的时效温度不足以消除基体的晶格畸变, 从而导致试样的析出顺序发生改变。

Hu Z. Y. 等^[79] 研究了喷射成形快速凝固细晶 Al-Cu-Mg 合金在快速冷冲及再结晶退火工艺过程中的微结构演变。不同道次快速冷冲 Al-Cu-Mg 合金试样再结晶退火后的析出相形貌 TEM 图如图 4 所示。由图可见, 合金的主要析出相为 S 相, 由于添加了一定的 Mn, 还可以观察到少量较粗大的 Al₆Mn 相。随着变形道次的增多, 试样变形量不断增加, 析出相密度不断增大、尺寸不断减小, 越趋于弥散分布。快速冷冲引入的缺陷有助于 Al-Cu-Mg 合金脱溶, 促

进 S 相的形核与长大。

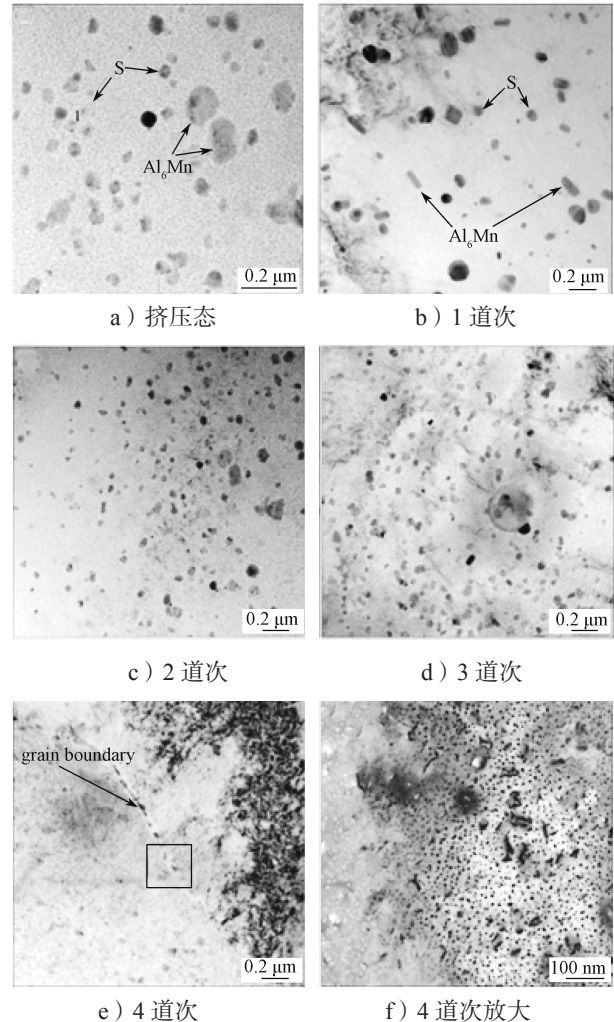


图 4 不同状态下快速冷冲 Al-Cu-Mg 合金试样再结晶退火后的析出相 TEM 图

Fig. 4 TEM images of the precipitation phases after recrystallization annealing of Al-Cu-Mg alloy specimens under different rapid cold stamping deformation

3 回溶和再析出对 Al-Cu 合金组织与性能的影响

回溶和再析出可使 Al-Cu 合金的晶粒细化, 从而一定程度上有效提高了 Al-Cu 合金的物理化学性能, 如: 硬度、抗腐蚀性能、抗冲击性能、抗拉强度和断后伸长率等。

赵凤晓等^[80] 研究了固溶原子及析出相对强变形 Al-4%Cu 合金晶粒细化效果的影响。室温下, 经相同变形道次后, 由于固溶原子的存在导致 Al-4%Cu 合金中亚晶内位错密度较高, 亚晶粒呈类竹节状; 纯

Al 中亚晶内位错密度低, 亚晶粒呈等轴状。室温下 Al-4%Cu 合金的析出相回溶抑制了细小晶粒的形成, 50 °C 下析出相回溶并再析出有利于细小晶粒的形成。Fan C. H. 等^[67]研究了快速冷冲变形中 Al-Cu-Mg 合金的硬度与加工道次的关系。不同道次快速冷冲变形中 Al-Cu-Mg 合金的硬度变化曲线如图 5 所示。由图可见, 第 1 道次和第 4 道次快速冷冲变形是硬度值增长最快的两个阶段, 第 2、3 道次合金硬度值略有增加, 但增长幅度小, 尤其是第 3 道次快速冷冲过程中合金硬度值还出现了小幅下降。挤压态 Al-Cu-Mg 合金在快速冷冲变形过程中硬度值得到显著提高, 由 55 HB 增加到 125 HB, 升高了约 127%。

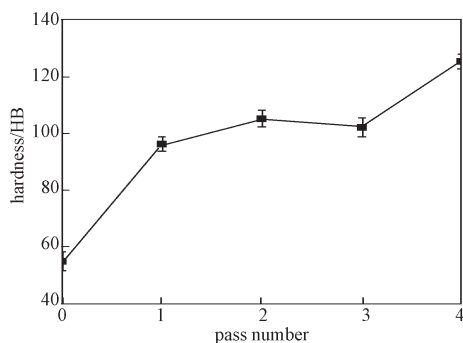


图 5 不同道次快速冷冲变形中 Al-Cu-Mg 合金硬度变化曲线

Fig. 5 Hardness of Al-Cu-Mg alloy as function of various passes of rapid cold stamping process

Meng Q. 等^[81]以 2060-T8 铝合金搅拌摩擦焊接头为研究对象, 研究了析出相演变对其显微组织和腐蚀行为的影响。结果表明, 腐蚀行为与各区析出相的特性密切相关, 由于 θ' 相、T1 相和 δ' 相的回溶, 以及晶界相和无沉淀析出带的出现, 轴肩影响区是腐蚀最敏感的区域; 因为不存在晶界析出相, 相比轴肩影响区, 热影响区的抗腐蚀性能略有提高。Zhang Z. G. 等^[82]以 Al-5%Cu 合金为研究对象, 经 ECAP 挤压后, 易被腐蚀的 θ 相被细化并固溶在基体中, 从而有效提高了合金的抗腐蚀性能。周伟等^[83]在 200 °C 退火 Al-4%Cu 合金中发现有少量第二相析出; 在 250 °C 退火后, Al-4%Cu 合金中析出大量针状纳米级 θ' 相, 并弥散分布于基体中, 显著提高了靶材的抗冲击性能, 断后伸长率从 3.5% 升高至 15.6%。何浩鹏等^[84]用中温 (150 °C) 强塑性变形研究方法, 诱导 2297-T87 铝锂合金析出相回溶至基体。在强变形停止后, 再时效处理过程中, 过饱和固溶体析出速度显著减慢, 析出序列不变, 经时效处理后铝锂合金的抗拉强

度与伸长率均高于原始试样, 合金热稳定性增强。

4 研究方向及发展趋势

本研究综述了 Al-Cu 合金在强塑性变形过程中纳米析出相的演变规律, 综合分析了析出相回溶的机理、机制及回溶和再析出对 Al-Cu 合金组织和性能的影响, 总结强塑性变形下 Al-Cu 合金纳米析出相回溶和再析出行为的研究方向及发展趋势概括为以下几个方面。

1) Al-Cu 合金回溶扩散过程中, 原子回溶到基体中的途径和方式, 以及在基体中形核和长大的机制还有待深入研究。

2) 研究 Sc、Zr、Ag 等微量元素对 Al-Cu 合金的强化机制和作用机理, 探讨多元微合金化对析出相回溶和再析出的影响机制, 以及多元微合金化对提升合金耐蚀性、强韧性及耐高温性能的影响机理是未来的重点研究方向。

3) 在强塑性变形条件下, 探讨形变能对 Al-Cu 合金纳米析出相的形貌、分布及与基体取向关系的影响也是未来重点研究方向之一。

4) Al-Cu 合金在长时间使用过程中, 其强度、塑性等相关物理化学性能的衰退速率和衰退机理需进一步探索研究。

随着科学技术的不断发展, 航空航天以及汽车工业等对高强钢 Al-Cu 合金材料的需求量显著增加, 深入探究 Al-Cu 合金纳米析出相回溶、再析出对合金高强韧性的影响机制意义重大。

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Current States and Development of Research on Redissolution and Reprecipitation of Nanoprecipitated Phases in Al-Cu Alloys

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Abstract: The evolution of nanoprecipitated phases in Al-Cu alloys under severe plastic deformation (SPD) was summarized. SPD at room temperature induced the precipitation of Al-Cu alloys to dissolve, leading to the reformation of supersaturated solid solution in the aluminum matrix. In the process of SPD or aging treatment after the SPD, the reprecipitated phases were precipitated from the aluminum matrix and the mechanical properties of the alloys were remarkably improved. The mechanism, system of the redissolution of precipitation phases and the effects of redissolution and reprecipitation on the microstructure and properties of Al-Cu alloys were comprehensively analyzed. The development and research direction of redissolution and reprecipitation of nanoprecipitated phases in Al-Cu alloys were also described in order to provide a theoretical reference for further application of Al-Cu alloys materials.

Keywords: Al-Cu alloy; nanoprecipitated phase; redissolution; reprecipitation