Microstructure evolution and densification of sintered porous 2024 aluminum alloy compressed in a semi-solid state

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Abstract

The semi-solid powder forming (SSPF) as a promising technology has a complicated process, but the formation mechanism of that remains unclear. The study of semi-solid compression of porous materials is an effective method to investigate the SSPF. Based on analyzing the influence of deformation strain, temperature, strain rate and initial relative density on microstructure evolution and relative density development of sintered porous 2024 aluminum alloy during semi-solid compression, the deformation mechanism and densification mechanism of semi-solid porous materials were proposed. The results show that main deformation mechanisms of porous materials during semi-solid compression are breakup of the solid skeleton or powders, and flowing of liquid with powders or fragments. Breakup mechanism is dominant at center area, whereas flowing mechanism dominates at the margin. With increasing strain and temperature and decreasing strain rate, breakup mechanism becomes primary. The densification mechanisms of porous materials during semi-solid compression are rearrangement of powders or fragments and filling of liquid, resulting from collapse of the solid skeleton or powders and flowing of liquid. The emergence of pores and densification occur simultaneously during semi-solid compression of porous materials.

1. Introduction

Wu et al (2010) considered the semi-solid powder forming (SSPF) is a new technology with advantages of powder metallurgy and semi-solid forming, which has been widely used in many fields. Li et al (2014) demonstrated the process of SSPF is attractive due to its conceptual simplicity and low cost. Although the SSPF has been investigated by many researchers, the deformation mechanism and densification mechanism of the SSPF still remain unclear. Because the SSPF is a complicated processing during which samples deform, solidify and are densified simultaneously. It is proposed by Luo et al (2014 and 2019). As the simple stress state and existence of pores during semi-solid compression of porous materials, some interesting phenomena (such as powder breakup that occurs certainly but has rarely been found during the SSPF) were observed, which are helpful to illustrate formation mechanisms of the SSPF. Therefore, the semi-solid compression of porous materials was used to help study the SSPF.

Tzimas and Zavaliangos (1999) considered that the deformation mechanism of semi-solid dense material is breakup of the solid skeleton. Chen and Tsao (1997) proposed four major mechanisms during semi-solid deformation of dense materials: liquid/flow(LF), flow of liquid incorporating solid particles (FLS), sliding between solid particles (SS), and plastic deformation of solid particles (PDS) mechanisms. Nagira et al (2011) considered that globule rearrangement involving dilation is the main deformation mechanism of globular

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semi-solid steel. Wu et al (2016) described the deformation behavior and characteristics of porous materials during semi-solid compression. However, investigations on the deformation mechanism of porous materials or powders compressed at a semi-solid state are scarce. Porous materials or powders during semi-solid compression also have liquid but that is inside powders, thus its deformation mechanism is different from that of the dense material.

Chen and Chen (2012) considered the main densification mechanisms of porous materials or powders during cold compaction are plastic deformation, particle rearrangement and little breakup of powders. Plastic deformation, powder restacking, power-law creep and various kinds of diffusion (grain-boundary diffusion, lattice diffusion, surface diffusion, and vapor transportation) are dominant densification mechanisms of porous materials or powders during hot pressing and sintering, as proposed by Atkinson and Davies (2000). German et al (2009) considered capillary force is the driving force of densification during liquid-phase sintering, the major densification mechanisms of which are powder rearrangement, filling of liquid, and diffusion. However, very few research efforts have been reported on densification mechanism of porous materials or powders compressed at a semi-solid state. There are pores existing in porous materials or powders during semi-solid compression too, thus its densification process is still the shrinkage or disappearance of pores. Therefore, semi-solid compression of porous materials or powders can draw lessons from cold compaction, hot pressing and liquid-phase sintering. Nevertheless, because of the presence of liquid, its densification mechanism is different from those of cold compaction and hot pressing. As external pressure is imposed on semi-solid porous materials or powders, its densification mechanism is also different from that of liquid-phase sintering.

In order to investigate deformation mechanism and densification mechanism of porous materials or powders during semi-solid compression, the sintered porous 2024 aluminum alloy was isothermally and uniaxially compressed among the semi-solid temperature range in this study. The porous material was sintered by Spark Plasma Sintering (SPS). The effects of deformation strain, temperature, strain rate and initial relative density on the microstructure evolution and relative density development were analyzed. Based on the above, deformation mechanism and densification mechanism of porous materials or powders during semi-solid compression were discussed.

2. Experimental

Gas-atomized spherical powders of commercial 2024 aluminum alloy (Al-4.09Cu-1.18Mg-0.54Mn-0.21Fe wt%, Beijing General Institute of Nonferrous Metals Research) were used in this study, the mean particle size of which is 53.5 μm. Powders in graphite mold with the inner diameter 30.4 mm were heated to the pre-set temperature at the rate of 50 °C min⁻¹ (below 400 °C) and 20 °C min⁻¹ (above 400 °C), and then held for 2 min, subsequently which were cooled in the chamber with about 40–60 min to 100 °C and then were taken out to cool in the air. 12 active DC pules with a pulse duration 3.3 ms were followed by 2 periods of inactive DC pluses with 0 current, which means that the pulse sequence is 12:2. Sintered porous specimens with relative densities of 63%, 68%, 83% and 90% were obtained at 300, 350, 400 and 440 °C, respectively, at vacuum by SPS-825 system. The pressing force applied on specimens sintered at 300 and 350 °C was 0.7 KN, while that at 400 and 450 °C was 3.5 KN. Compression samples with a size of φ8 mm × H12 mm were taken from the center of specimens by wire-electrode cutting. Then samples with aluminum-clad films (thickness 0.2 mm) were compressed in a semi-solid state with Gleeble-350 thermal simulation testing machine. In order to study the microstructure evolution and relative density evolution, compression samples were heated to 580, 590 and 600 °C, respectively, held for 5 min and then compressed with the strain of 20%, 50% and 80%, respectively, at the strain rate of 0.1, 1 and 5 s⁻¹, respectively. The liquid mass fraction of 2024 aluminum alloy at 580, 590 and 600 °C is 11.3%, 15% and 19.9%, respectively, which was calculated by DSC method and indicated at details in the article (Wu et al (2017)). Samples after compression were immediately quenched in water to keep the microstructure characteristics in a semi-solid state. Afterward, samples were polished and corroded with 10 s by Keller’s agent (95 mLH₂O + 2.5 mLHNO₃ + 1.5 mLHCl+1.0 mLHF, Tamadon et al (2017)). The microstructure was observed by LcicaDM1500M. SEM images were obtained by Quanta2000 at secondary electron mode with voltage of 20 kV. The sintered density of samples was determined by using Archimedes’ method with distilled water at room temperature as test medium, which was the average value of three measurements. The relative density was calculated by the mean sintered density/theoretical density × 100, while the theoretical density of the 2024 Al alloy is 2.78 g cm⁻³.
3. Results

3.1. Microstructure evolution

3.1.1. The influence of strain on microstructure evolution

Seen from figures 1(a) and (b), the central and marginal powders of samples remain their original spherical shapes when the strain is 20%. As shown in figures 1(c) and (d), particles are fine and few spherical powder boundaries are observed at the center of samples, but which are largely observed at the margin. Hence, it is illustrated that most of central powders are broken up into fragments, whereas most of marginal powders keep the spherical shape, as the strain increases to 50%. Particles in figures 1(e) and (f) are smaller and distribute more closely than those in figures 1(c) and (d). It is deduced that powders at the center and edge of samples all crush into fine particles, as the strain keeps increasing to 80%.

Therefore, it can be concluded that more central and marginal powders of samples collapse into fragments with increasing strain, making particles finer. It is consistent with semi-solid powder rolling. Liu et al. (2014) demonstrated grains of strips gradually become finer when strips are rolled from sections 1 to 4 during semi-solid powder rolling.
3.1.2. The influence of temperature and initial relative density on microstructure evolution

The microstructure at the center of the sample compressed at 590 °C (figure 2(a)) is similar to that compressed at 580 °C (figure 1(c)). There are some round pores in powders at the margin of the sample (figure 2(b)). As compared with figures 2(a) and (c), central particles of the sample compressed at 600 °C are larger and rounder. Seen from figures 2(b) and (d), there are few spherical boundaries at the edge of the sample compressed at 600 °C. Therefore, it is concluded that more central and marginal powders of samples crush into fragments and those become coarse, as temperature increases. It is similar with the result of sintered porous AA2024 compressed at 580 and 600 °C with initial relative density of 83%, which was investigated by Wu et al (2017). Thus, it indicates that the initial relative density has few effects on the microstructure evolution.

3.1.3. The influence of strain rate on microstructure evolution

As shown in figures 3(a), (c) and 1(c), central particles of samples compressed at a lower strain rate are finer, whereas there are more spherical powders at a higher strain rate. Seen from figures 3(b), (d) and 1(d), the microstructure evolution at the margin of samples has the similar trend with that at the center, except for more spherical boundaries. Therefore, it infers that there are more powders crushing at a lower strain rate, whereas more powders flowing at a higher strain rate.

3.2. Relative density development

Figure 4(a) shows the average relative density of sintered porous 2024 aluminum alloy with different initial relative densities compressed to the strain of 50%, at 580, 590 and 600 °C, respectively. It is the mean value of relative densities obtained at the strain rate of 0.1, 1, and 5 s⁻¹, respectively (with other conditions unchanged). As seen from figure 4(a), a higher relative density is obtained at a higher temperature, no matter what initial relative density is. It is because more powders or fragments fill pores, resulting from more powders breaking up at a higher temperature (as discussed at section 3.1.2). Besides, the coarsening of particles also reduces pores.
Figure 4(b) shows the average relative density at the strain rate of 0.1, 1, and 5 s$^{-1}$, respectively. It is the mean value of relative densities obtained at 580, 590 and 600 °C, respectively (with other conditions unchanged). As shown in figure 4(b), relative density decreases with increasing strain rate. The reason is that there is more time for powders to crush at a lower strain rate, leading to more powders or fragments filling pores.

The value of the ordinate in figure 4(c) is the difference between average relative density (in figure 4(b)) and initial relative density. Seen from figure 4(c), the difference firstly increases and then decreases below zero as the initial relative density rises. In other words, samples with low initial relative densities become dense, while the relative density of samples with high initial relative densities declines after the semi-solid compression. It relates to the emergence of new pores, which will be discussed at details in section 4.2.

4. Discussions

4.1. Deformation mechanism

Based on the above experimental results, powders at the center of porous materials compressed at a semi-solid state collapse into fragments, and powders at the margin keep the initial spherical morphology and flow with liquid. With decreasing strain rate and increasing strain and temperature, more powders crush into pieces. In consequence, it can be deduced that deformation mechanism of semi-solid porous materials during the uniaxial compression is related to breakup of powders and flowing of liquid. Compared with dense materials, the crushing of solid skeleton will also take place for porous materials. Besides, deformed or flatten powders and grains are not observed in all the optical micrographs, which infers that plastic deformation may hardly occur during semi-solid compression.

Consequently, the main deformation mechanism of porous materials during semi-solid compression is breakup and flowing. The breakup consists of the solid skeleton collapsing and powders crushing. The collapsing of solid skeleton is due to the sliding of powders along sintered necks between powders (shown in figure 5), which results from the lubrication of liquid. Figure 5 is the SEM image of powders that are squeezed out of the side-surface of a sample after semi-solid compression. It is obviously seen that powders are connected...
by sintered necks (arrow-marked deep color area). At the surface of powders, there are a deep color area (circled out in figure 5) occupied previously by sintered neck, and a saddle-backing that is the elongated sintered neck. It is verified that liquid exists at sintered necks and powders slide along sintered necks, making the solid skeleton collapse.

Figure 4. The average relative density (ARD) of sintered porous 2024 aluminum alloy compressed to the strain of 50% at a semi-solid state: (a) temperature versus ARD curves with different initial relative densities, (b) strain rate versus ARD curves with different initial relative densities, (c) initial relative density versus the difference (between ARD and initial relative density) curves under different strain rates.

Figure 5. SEM of powders connected by sintered necks at the side-surface of a sample after semi-solid compression (68%, 600 °C, 1 s⁻¹).
The crushing of powders is due to the sliding of grains inside powders, which results from liquid lubricating grain boundaries. It is seen from figure 6(a) that there are clear boundaries between grains in a single powder. The round concave surface of the broken powder in figure 6(b) is smooth. It infers that grains slide out of the powder along grain boundaries. It also explains the existence of some round pores in powders (shown in figures 2(b) and 3(b)).

The flowing includes liquid flowing and the powders or fragments flowing incorporated with liquid. Much liquid is entrapped inside powders, which can flow only when it is squeezed out of powders. Hence, only a small part of liquid in porous materials runs individually. There are solidified liquid-phase bridges at the side-surface of a sample after semi-solid compression (shown in figure 7(a)). Figure 7(b) is the amplified image of figure 7(a). It can be clearly seen that the liquid wraps powders or pieces and flows with them. Thus, a large part of liquid in porous materials flows with powders or fragments. It is deduced that the breakup of powders and the flowing of liquid (together with powders or pieces) should also occur during the SSPF, as its external force is far larger than that in a semi-solid compression to make powders easier to crush.

The ratio of breakup and flowing is variant at different regions of samples and compression conditions. The center area of samples is dominated by breakup mechanism, while flowing mechanism is dominant at the margin. As inferred from section 3.1, with increasing strain (and temperature) and decreasing strain rate, the breakup mechanism becomes principal. Based on experimental results and discussions above, the schematic
diagram of deformation for porous materials during semi-solid compression is shown in figure 8. As seen from figure 8(a) (Luo et al. 2019), the porous sample is consisted of powders (blue or dark areas) which are connected with sintered necks (black areas), and there are liquid pockets (yellow or light areas) inside powders and sintered necks. As shown in figure 8(b), the number of sintered necks reduces, which means that the solid skeleton crushes. Afterward, some central powders collapse into fragments and liquid flows out from broken powders, which will fill in gaps or pores between powders. Powders run to the margin simultaneously. When powders contact with each other (in figure 8(c)), they further rupture by sliding along grain boundaries under the applied pressure, and liquid with fragments and powders flows to the sample surface.

4.2. Densification mechanism

The plasticity theory proposed by Shima and Oyane (1976) and Doraivelu et al. (1984) is widely used for cold compression or hot pressing of porous metals and powders. It is considered that the yield criterion is a function of the first invariant of stress and the second invariant of deviatoric stress, and the strain-stress relation follows the flow rule. Because $\sigma_2 = \sigma_3 = 0$ and $d\varepsilon_2 = d\varepsilon_3$ in the uniaxial compression, axial strain and relative density have relations as following, which are derived from Shima and Doraivelu yield criteria. For Shima yield criterion

$$d\varepsilon_1 = -\frac{d\rho (1 + 9f^2)}{3\rho}$$

(1)

where $\varepsilon_1$ is the axial strain, $\rho$ is the relative density after compression, $f$ is the material-related parameter which is related to the relative density. For aluminum alloy, $f$ is mostly represented by

$$f = \frac{1}{2.5(1 - \rho)^{0.5}}$$

(2)

By setting $\varepsilon_1 = 0$ at $\rho = \rho_0$, integration of equations (1) and (2) results in

$$-\varepsilon_1 = 0.813 \ln \frac{\rho}{\rho_0} + 0.48 \ln \frac{1 - \rho}{1 - \rho_0}$$

(3)

For Doraivelu yield criterion

$$d\varepsilon_1 = -\frac{d\rho}{\rho(1 - \rho^2)}$$

(4)

By setting $\varepsilon_1 = 0$ at $\rho = \rho_0$, integration of equation (4) results in

$$-\varepsilon_1 = \ln \frac{\rho}{\rho_0} + \frac{1}{2} \ln \frac{1 - \rho_0^2}{1 - \rho^2}$$

(5)

The relative density after compression in figure 9 was calculated by equations (3) and (5), when the strain $\varepsilon_1$ is 50% and initial relative densities are 63%, 68%, 81% and 90%, respectively. The calculated values are larger than experimental results obtained at different temperatures and strain rates. It infers that the densification mechanism of porous materials under semi-solid compression is different from those under cold compaction and hot pressing. Similar with them, rearrangement and sliding of powders also occur during semi-solid
compression of porous materials. But the evidence of plastic deformation is not obvious during the process. Thus the contribution of rearrangement and sliding is smaller than that of plastic deformation for densification. Besides, the collapse of skeleton and powders and the flowing of powders and liquid (as discussed in section 4.1) make the height of samples reduce and the diameter increase, leading to partial densification.

In contrast with liquid-phase sintering, porous materials during semi-solid compression are densified under an applied force, which is much greater than a capillary force. In addition, porous materials compressed in a semi-solid state don’t have enough time to creep and diffuse, as compression time is short in this study. Only when the strain rate is below 0.1 s$^{-1}$, creep and diffusion may occur. But there is no obvious evidence in this work to prove their existence. Luo et al (2015) and Luo and Liu (2016) considered that liquid flowing, particle rearrangement and restacking happen during the first section of the SSPR, while the main densification mechanism of second and third section is plastic deformation. Compared with SSPR, plastic deformation of powders is not observed in this job. Its reason is that the semi-solid compression of porous materials is an isothermal process, resulting in much liquid.

On the base of experimental results and theoretical analysis above, the deformation mechanism (breakup and flowing) of porous materials during semi-solid compression contributes to densification. The collapsing of solid skeleton releases more pores and powders, which are rearranged under an applied pressure to make pores converge and disappear (shown in figure 8(b)). Powders crush into pieces which are restacked and fill in pores (shown in figure 8(c)). The flowing of liquid also results in the rearrangement of powders or fragments and the filling of liquid, which can bring pores to the side-surface. In a word, the densification mechanism of porous materials during semi-solid compression is restacking of powders or fragments and filling of liquid, resulting from breakup and flowing. When the temperature increases and strain rate decreases, the rearrangement mechanism of powders or pieces becomes dominant, resulting from the dominant deformation mechanism of powder crushing.

However, the liquid flowing at a semi-solid state will certainly cause the emergence of pores. Karch et al (2014) considered there are many micro-pores in the dense materials after semi-solid compression. Lewandowski and Overfelt (1999) demonstrated the reason of micro-pores appearance is that no much liquid is immediately supplied to fill the vacancy, caused by liquid running away (along grains boundaries) and the volume shrinkage during solidification. The liquid flowing exists throughout the whole semi-solid compression process of porous materials (as discussed section 4.1). Consequently, pores are certain to appear during semi-solid compression of porous materials. Additionally, the breakup of solid skeleton and powders during semi-solid compression will also result in new pores forming. As seen from figure 10, pores (circled out) in a broken powder are caused by liquid flowing. Pores (arrow-marked) in triangle regions outside the broken powder are caused by powders crushing and then rearranging.
The initial relative density of samples will affect the formation of pores, as discussed in section 3.2. The initial relative densities of compression samples are 63%, 68%, 83% and 90%, respectively, while the liquid mass fraction of 2024 aluminum alloy at 600 °C is 19.9%, respectively. Thus, it is obviously seen that there are not enough spaces in samples with higher initial relative densities to be filled in by liquid. Consequently, liquid may be squeezed out of samples and their mass is reduced, while their volume is not changed because of initial little pore spaces. As a result, the relative density will be decreased. Moreover, the flowing liquid (to the sample surface) also makes new pores emerge and the relative density decrease in consequence, as discussed above.

Besides, in contrast with other aluminum alloys, 2024 at a semi-solid state is more likely to generate pores because of the large freezing range, which was proposed by Liu et al. (2005). Wu et al. (2016) measured that the freezing range of 2024 aluminum alloy powders is 139.3 °C, which is larger than those of A356 and Al6061. Hu et al. (2016) measured that of A356 is 63 °C, while Kim et al. (2007) illustrated it is 62 °C. Wu and Kim (2015) tested the freezing range of Al6061 is 110 °C. Thus, the liquid fraction increment of 2024 aluminum alloy is slower than those of A356 and Al6061 with the increasing temperature, which may lead to less liquid filling and more pores formation.

Therefore, the emergence of pores and densification occur simultaneously during semi-solid compression of porous materials. When the initial relative density is high, the emergence of pores is dominant. Consequently, full densification of porous 2024 aluminum alloy will not be achieved during semi-solid uniaxial compression, which means that the degree of densification is not high. For semi-solid powder forming, it is better under a bi-axial or tri-axial stress state to achieve full dense materials.

5. Conclusions

1. As strain increases and strain rate decreases, particles at the center of porous materials compressed at a semi-solid state become finer and denser, whereas fewer powder boundaries are observed at the margin. The initial relative density has few effects on the microstructure evolution.

2. As temperature increases and strain rate decreases, the relative density of porous materials compressed at a semi-solid state increases. As the initial relative density increases, the difference between relative density and initial relative density firstly increases and then decreases.

3. The major deformation mechanisms of porous materials during semi-solid compression are breakup of the solid skeleton or powders and flowing of liquid with powders or fragments. Breakup mechanism is dominated at center area, whereas flowing mechanism is dominant at the margin. As strain and temperature increase and strain rate decreases, the breakup mechanism becomes dominant.
4. The densification mechanisms of porous materials during semi-solid compression are the rearrangement of powders or fragments and the filling of liquid, which owe to the deformation mechanisms. As strain and temperature increase and strain rate decreases, the rearrangement mechanism of powders or fragments becomes dominant.

5. The emergence of pores and densification occur at the same time during semi-solid compression of porous materials. The flowing of liquid, breakup of the skeleton or powders, high initial relative density and the large freezing range are main reasons for pore formation. Full densification of porous 2024 aluminum alloy will not be achieved during semi-solid uniaxial compression, which may be achieved under a bi-axial or tri-axial stress state during semi-solid forming.

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Data availability statement

No new data were created or analysed in this study.

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