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ARTICLE

Comparison of Discontinuous Yielding Phenomenon and Adiabatic Temperature Rising Effect of Ti-5553 Alloys Prepared by Powder Metallurgy and Ingot Metallurgy

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Abstract: Discontinuous yielding phenomenon (DYP) and adiabatic temperature rising (ATR) effect were investigated for Ti-5553 (Ti-5Al-5Mo-5V-3Cr) alloys prepared by powder metallurgy (PM) and ingot metallurgy (IM) approaches during hot compression testing conducted at the temperature range of 700~1100 °C and the strain rate range of 0.001~10 s^{-1}. The results show that the magnitude of yield drop exhibits a positive correlation to strain rate but nearly a negative correlation to deformation temperature for both PM and IM alloys, and the occurrence of DYP in the alloys is elucidated by the dynamic theory. IM alloy shows a higher degree of yield drop than PM alloy under the same condition because of low initial dislocation density in as-cast state and the subsequently promoted newly-generated mobile dislocation from grain boundary. A strong positive correlation between ATR effect and strain rate but intensive negative correlation between ATR effect and deformation temperature are discovered for the two alloys. PM alloy shows a lower degree of ATR effect under the same processing condition than IM alloy as a result of its lower deformation resistance and higher deformation compatibility.

Key words: Ti-5553 alloy; powder metallurgy; hot deformation; discontinuous yielding phenomenon; adiabatic temperature rising

Ti-5553 alloy is a kind of metastable β titanium alloy and has already been used for aerospace applications (like the landing gears for Boeing-787 and Airbus-350 aircraft[1,2]) extensively thanks to its balanced mechanical properties, especially the ultra-high-strength, excellent hardenability and great fatigue resistance. However, the high manufacturing and processing cost of titanium alloys restrict their further applications in chemical, medical and civil fields. Powder metallurgy (PM) approaches are widely regarded as feasible and effective ways to overcome the cost-affordable problem of titanium products compared to the conventional ingot metallurgy (IM) approaches by distinct advantages like low-temperature manufacturing and near-net-shape forming, which reduce the preparation and processing cost significantly. Ti-5553 alloy has relatively low processing temperature and high oxidation resistance so it is also suitable for manufacturing small-sized PM aircraft and marine parts[3].

Thermomechanical processing (TMP, like forging, extrusion and rolling) is essential for producing qualified metastable β titanium alloy products, nevertheless, it is difficult to process titanium alloys because of their intrinsic crystalline structure and high sensitivity to the processing variables. As a result, there are extensive works that report the hot deformation behavior of metastable β titanium alloys, while the related researchers are mainly focused on the establishment of processing maps, microstructural evolution processes and deformation mechanism of the studied alloys. Other important and interesting phenomena/effect like discontinuous yielding (DYP) and adiabatic temperature rising (ATR) is seldom reported for metastable β titanium alloys.

Different from continuous yielding, DYP refers to considerable yield drop after the peak stress appears at the end of the initial work hardening stage of the alloy during deformation, which has a significant influence on the deformation

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response of the alloy that undergoes TMP. There are some works conducted on DYP for metastable β titanium alloys. Zhou et al[4] investigated DYP of heat-treated (IM) Ti-5553 alloy, and discovered that DYP of the alloy is promoted at high-strain-rate deformation rather than at low-strain-rate deformation. Fan et al[5] found that DYP in processed (IM) Ti-7333 alloy is facilitated at a lower strain rate and higher temperature, and the strain rate plays a more important role. It is clear that the effect of deformation parameters on DYP of metastable β titanium alloys is still under debate, and there is little work conducted for PM titanium alloys.

ATR is a common phenomenon during TMP of metallic materials due to external stress and work especially for titanium alloy at high-strain-rate deformation as its low thermal conductivity. ATR effect can be advantageous to the hot processing of the working piece in some situation for the retaining of desired processing temperature for a longer time. However, serious ATR will lead to adverse effects on the hot processing of the parts with the reduction of the materials' hot workability, including the formation of adiabatic shear banding (ASB)[6,7], thermal cracking and unexpected microstructure variation. Meanwhile, ATR can be associated with the kinetic analysis and influence the mechanical behavior of the working pieces, and the temperature deviation can be generated to change the actual TMP condition as well. Whereas, there are seldom systematic investigations about ATR for metastable β titanium alloys, let alone the PM ones.

In this work, the DYP and ATR for PM and IM Ti-5553 alloys were investigated simultaneously to offer comparative research. The effect of alloys' initial microstructure, processsing history and deformation parameters on these two phenomena was discussed comprehensively to reveal the underlying mechanisms and to provide the theoretical basis for hot processing of PM and IM metastable β titanium alloys.

1 Experiment

The PM Ti-5553 alloy in this study was synthesized using a powder mixture with the nominal composition of Ti-5Al-5Mo-5V-3Cr (wt%) prepared from elemental powders of pure-Ti (hydride-dehydride, HDH), pure-Al and master alloy powders of V-Al (65 wt%~35 wt%), Mo-Al (85 wt%~15 wt%) and Cr-Al (70 wt%~30 wt%). The powder mixture (500 g) was warm-pressed into a cylinder green powder compact at about 250 °C under uniaxial pressure of about 400 MPa in air. After that, the powder compact was consolidated into alloy billet (diameter of 58 mm, relative density of 98%) by the modified hot-pressing process, which is described in Ref.[8]. The used IM Ti-5553 alloy billet was obtained from a 35 kg as-received ingot manufactured through conventional double vacuum arc remelting (VAR) and casting. The measured chemical composition of PM (as-consolidated) and IM (as-cast) Ti-5553 alloys is displayed in Table 1. PM alloy has a similar chemical composition to IM alloy in main alloying elements, but much higher oxygen content (0.36 wt% and 0.08 wt%).

Table 1 Actual chemical composition of PM and IM Ti-5553 alloy (wt%)

Alloy	Al	Mo	V	Cr	O	Ti
PM	4.99	4.94	4.93	2.90	0.36	Bal.
IM	5.14	5.02	5.03	3.10	0.08	Bal.

The initial microstructures of PM and IM Ti-5553 alloys are shown in Fig.1. As shown in Fig.1, both PM and IM alloys have the typical β grain matrix with precipitated phases. IM alloy possesses a large number of dispersed α phases spreaded over β matrix, while PM alloy only possesses a small number of agminated precipitates (α and α'') distributed along β grain boundaries[9]. Moreover, much finer grain can be found in the PM alloy instead of IM alloy, with the grain size of 100 and 1000 μm, respectively. Particularly, some residual pores can be identified in the microstructure of PM alloy. The β phase transformation temperature of PM and IM alloys are determined metallographically as 975±5 °C and 875±5 °C, respectively[10].

Gleeble® 3800-GTC thermal physical simulator was employed to perform the hot compression of the alloys in the temperature range of 700~1100 °C (100 °C interval) and strain rate range of 0.001~10 s^{-1}, with the deformation degree of 70% sample's height reduction. Graphite foils and Tantalum sheets were used to improve conductivity and to reduce the friction of the testing system. Cylinder specimens were cut from the alloy billets and machined to a target size of 10 mm in diameter and 15 mm in height. The specimens were heated to the preconcerted temperatures (at a heating rate of 10 °C/s) and then kept

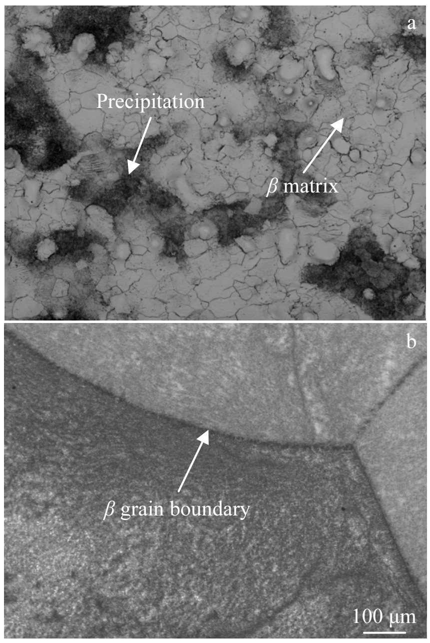

Fig.1 Initial microstructures of PM (a) and IM (b) Ti-5553 alloys

for 4 min to obtain homogeneous temperature distribution throughout the specimen. After the compression testing in vacuum, all the specimens were water-quenched to freeze the deformed microstructure. Furthermore, some deformations were stopped at the true strain of 0.02, for the microstructure examination of the slightly-deformed specimen. In the course of hot compression, the stress-strain relationship and the actual temperature of the specimens were recorded by the system automatically. The room-temperature tensile tests were conducted using an Instron-5982 universal machine at a strain rate of 1×10^{-3} s^{-1}. The rectangle cross-section tensile specimens with the dimension of 4 mm×2.5 mm were cut from both PM and IM Ti-5553 alloy billets and gauge length was 15 mm.

The metallographic specimens were ground, polished and then etched using Kroll's reagent (10 mL HF+20 mL HNO_3+70 mL H_2O) for microstructure examination by OLYMPUS/PMG3 optical microscope (OM). For transmission electron microscopy (TEM) examination, the samples were ground down to 60 μm, and then the PM alloy specimens were ion milled and the IM alloy specimens were twin-jet electron polished for the observation by a JEM-2100 TEM facility.

2 Results and Discussion

2.1 Room-temperature tensile properties

Table 2 shows the tensile properties of PM and IM Ti-5553 alloys at room temperature. As exhibited in the Table, PM alloy shows the ultimate tensile strength (UTS) of 1008 MPa and the elongation to fracture of 2.1%, while IM alloy shows the UTS of 1215 MPa and the finial elongation of 5.8%. PM alloy shows both lower tensile strength and ductility than IM alloy. The relatively small size (500 g billet) PM alloy was consolidated by fast hot-pressing approach from powder compact at high temperatures of 1250~1300 °C, and then cooled down in flow argon at a high cooling rate due to the small size of the billet and the cool environment. The PM alloy is supersaturated after the hot pressing and fast cooling down to room temperature. The supersaturated microstructure not only contains the non-equilibrium α (only a small amount of α) and β phases, but also contains a higher dislocation density and internal stress (higher than IM alloy) due to the fast-consolidation by pre-deformation (hot-pressing), which makes the PM alloy weak and brittle at room temperature. Moreover, the residual pores in the microstructure and the high oxygen content of PM alloy also contribute to its lower ductility. Conversely, the large size (35 kg ingot) IM alloy is cooled down slowly (relatively) after arc-melting, and a larger number of α precipitates and bigger grain size can be observed in its initial microstructure, indicating high microstructural equilibrium of IM alloy. Therefore, the higher microstructural equilibrium, higher relative density (no residual pores), lower oxygen level and larger amount of α precipitation lead to the higher tensile strength and ductility of IM alloy collectively.

Table 2 Tensile properties of PM and IM Ti-5553 alloy at room temperature

Alloy	Yield strength/MPa	Ultimate tensile strength/MPa	Elongation/%
PM	935	1008	2.1
IM	1117	1215	5.8

2.2 Discontinuous yielding phenomenon

Fig.2 shows the true stress-true strain curves and the detailed yield drop situation after the peak stress of the PM and IM alloy compressed at 900, 1000 and 1100 °C and various strain rates from 0.001 s^{-1} to 10 s^{-1} (no DYP is observed at the temperature of 700 and 800 °C for two alloys at any stain rate). Moreover, the stress drop values under varying conditions are also exhibited in Table 3 to more clearly show the variation tendency of DYP.

From Fig.2 and Table 3, it is clear that the magnitude of DYP is varied with the deformation parameters and the discrepancy between PM and IM alloys is considerable. Typically, in most relative literatures about titanium alloys, the DYP has a positive temperature and strain rate sensitivity of the studied alloys[11,12]. That is to say, the DYP will be more obvious with increasing the deformation temperature and strain rate. Whereas, this tendency is not observed with complete identities in the PM and IM alloy investigated in this work. For the effect of strain rate, it is clear that DYP is roughly more obvious at high-strain-rate deformations rather than low-strain-rate deformations for the two alloys at different temperatures. However, in terms of temperature, the two alloys show nearly negative temperature sensitivity. Obvious DYP can be observed at 0.1~10 s^{-1} deformation rate when the temperature is 900 °C, while DYP is found only at very high-strain-rate deformation of 10 s^{-1} at 1000 and 1100 °C, accompanied by the reduced yield dropping magnitude as well.

The occurrence of DYP in metallic materials can be mainly explained by two mature theories. (1) Static theory (Fig.3a), which is associated with the dislocation pinning and rebooting[5,13]. In this theory, the dislocation movement is considered to be pinned by the solution atoms and/or impurities during hot deformation, and the loosening and rebooting of the dislocation from their pinning points are achieved when the external force reaches the certain critical value, which reflects the sudden yield drop in the flow curves from macroscopic view. (2) Dynamic theory (Fig.3b), which is associated with the sudden generation of the mobile dislocations from prior grain boundary[14,15]. In this theory, the dislocation density is suddenly increased when the hot deformation reaches the critical degrees, followed by the dislocation spreading to the grain interior with increasing the deformation strain. Although the static theory has been used to explain the DYP in some titanium alloys[5], it is hard to interpret the effect of deformation temperature and strain rate on DYP properly of the two studied alloys. Moreover, it may be difficult to pin the dislocation at such high-temperature deformation (such as 1100 °C) by solution atoms and/or impurities in the two alloys, which was also disapproved by

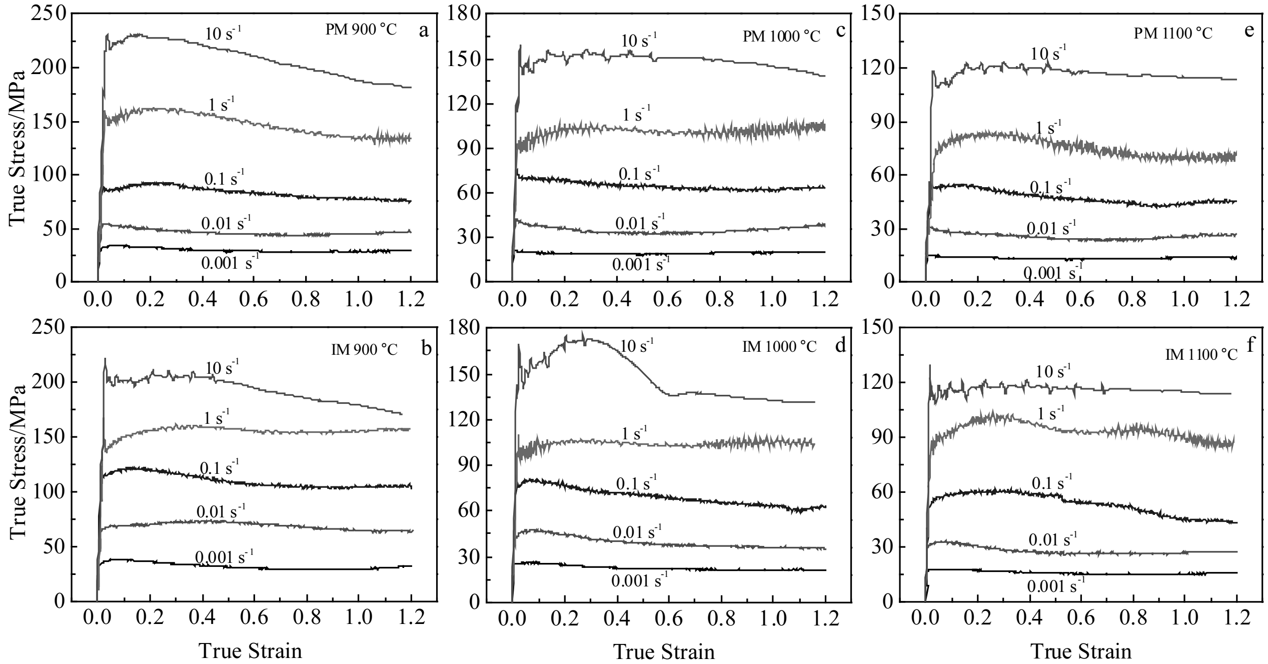

Fig.2 True stress-true strain curves of PM (a, c, e) and IM (b, d, f) Ti-5553 alloys under various conditions: (a, b) 900 °C, (c, d) 1000 °C, and (e, f) 1100 °C

Table 3 Yield drop values of PM and IM alloys deformed under varying conditions

Strain rate/s^{-1}	900 °C		1000 °C		1100 °C	
	PM/MPa	IM/MPa	PM/MPa	IM/MPa	PM/MPa	IM/MPa
10	14.20	22.18	18.95	29.95	9.7	21.63
1	14.39	30.95	/	17.26	/	/
0.1	6.71	12.22	/	4.31	/	/

Note: "/" refers no DYP (0 MPa) under specific condition; no DYP was observed at the strain rates of 0.01and 0.001 s^{-1}

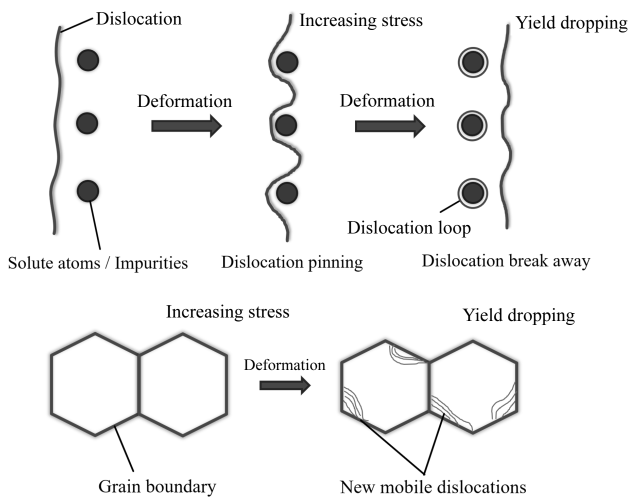

Fig.3 Schematic drawing of developed DYP mechanism of metallic materials during hot deformation: (a) static theory and (b) dynamic theory

Ankem et al[16] and Weiss et al[17]. Therefore, the DYP of the two studied alloys can be ascribed to the dynamic theory instead of the static one.

According to the dynamic theory, the prerequisite of DYP is that there is adequate dislocation density at the grain boundary, which can be realized by high strain rate and enough deformation strain. As a result, the increasing strain rate can obviously enhance the DYP of the two alloys by faster dislocation accumulation and generation, and there is no DYP at 0.01 and 0.001 s^{-1} deformations at all temperatures. When the deformation temperature is increased from 900 °C to 1000 and 1100 °C, the DYP is gradually weakened primarily due to the characteristic change of grain boundary and dislocation movement. This negative temperature sensitivity at high-temperature deformations has also been reported in β titanium alloys by Li et al[18] for Ti-3Al-5V-5Mo alloy and Vijayshankar et al[19] for Ti-Mn alloys.

The grains will suffer significant grain coarsening, which will lead to remarkable reduction in grain boundary density of the alloy. As demonstrated in the dynamic theory, the grain boundary acts as the source for the newly-formed mobile dislocations, so the reduced grain boundary density may cut down the dislocation source for DYP, resulting in the reduction of yielding drop magnitude. Furthermore, when the deformation is processed at higher temperatures, the subsequent higher driving force can promote the dislocation movement and intense dynamic softening (by dynamic recovery, dynamic recrystallization, etc), which consumes the accumulated dislocation notably, and then leads to the weakening of DYP.

Most importantly, it can be obviously observed from Fig.2 and Table 2 that IM alloy shows a higher degree of DYP than PM alloy under the same processing condition, indicating that PM alloy has higher flow stability than IM alloy during high-strain-rate deformation. Fig.4a and 4b show the initial TEM microstructure of PM and IM alloy, respectively. It is clear that PM alloy has a higher dislocation density than IM alloy before deformation, which is induced by the hot-pressing and subsequent fast cooling processes during the low-cost powder consolidation. After deformation at 900 °C/1 s^{-1} to the true strain of 0.02 (in the vicinity of yielding point), distinct dislocations initiating from grain boundary are observed for PM alloy (Fig.4c), while a larger number of dislocations generated from the β grain boundary are witnessed in IM alloy (Fig.4d). Hereinafter, the less significant DYP of PM alloy can be ascribed to its higher initial dislocation density. The relative higher dislocation density in the microstructure can hinder the sudden generation of new mobile dislocation for DYP during further deformation, then lowering the magnitude of the discontinuous yield drop. On the contrary, IM alloy has very low dislocation density in as-cast state, which provides a better precondition for the sudden origination of the mobile dislocations from grain boundary. Meanwhile, Fig.4c and 4d also verify that DYP in the two alloys complies with the dynamic theory. Furthermore, the chemical composition and initial microstructure also contribute to the lower yield drop magnitude. The higher oxygen content in PM alloy can introduce more solute atoms into the crystalline structure, which hinder the dislocation movement from grain boundary. As for the initial microstructure, finer grains can impede the sudden generation and extension of mobile dislocation as well.

2.3 Adiabatic temperature rising effect

Table 4 and Table 5 show the maximum temperature rising value of PM and IM alloy during thermal physical simulation at different temperatures (700~1100 °C) and strain rates (0.001~10 s^{-1}), respectively. Moreover, the three-dimension maps demonstrating the ATR distribution with varying the deformation parameters are exhibited in Fig.5. From the Tables and the maps, it is obvious that processing variables have a significant effect on ATR for both PM and IM alloys, and the ATR discrepancy between the two alloys is also considerable.

There is a strong positive correlation between ATR value and strain rate but an intensive negative correlation to deformation temperature for both the two alloys. Almost no ATR is observed at the deformation strain rates lower than 1 s^{-1} and the temperatures higher than 900 °C. The higher strain rate can provide more external energy and restrict the deformation

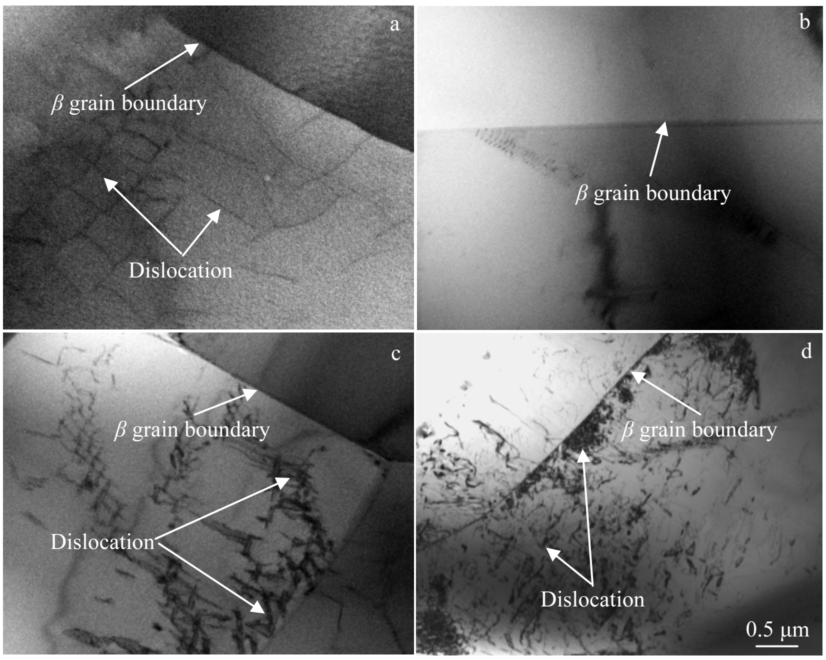

Fig.4 TEM images of Ti-5553 alloys before (a, b) and after (c, d) deformation at 900 °C/1 s^{-1} to a true strain of 0.02: (a, c) PM alloy and (b, d) IM alloy

Table 4 Maximum temperature rising value (°C) of PM Ti-5553 alloy during hot deformation at various temperatures and strain rates

Strain rate/s^{-1}	700 °C	800 °C	900 °C	1000 °C	1100 °C
10	68.4	46.0	33.2	18.5	6.6
1	26.2	20.1	10.9	5.5	2.4
0.1	9.2	6.4	4.6	/	/
0.01	/	/	/	/	/
0.001	/	/	/	/	/

Note: "/" refers no ATR (0 °C) under the specific condition

Table 5 Maximum temperature rising value (°C) of IM Ti-5553 alloy during hot deformation at various temperatures and strain rates

Strain rate/s^{-1}	700 °C	800 °C	900 °C	1000 °C	1100 °C
10	79.2	56.5	37.6	24.3	10.0
1	47.8	28.4	11.3	6.8	3.6
0.1[1]	13.0	9.5	6.8	2.8	/
0.01	2.5	/	/	/	/
0.001	/	/	/	/	/

Note: "/" refers no ATR (0 °C) under the specific condition

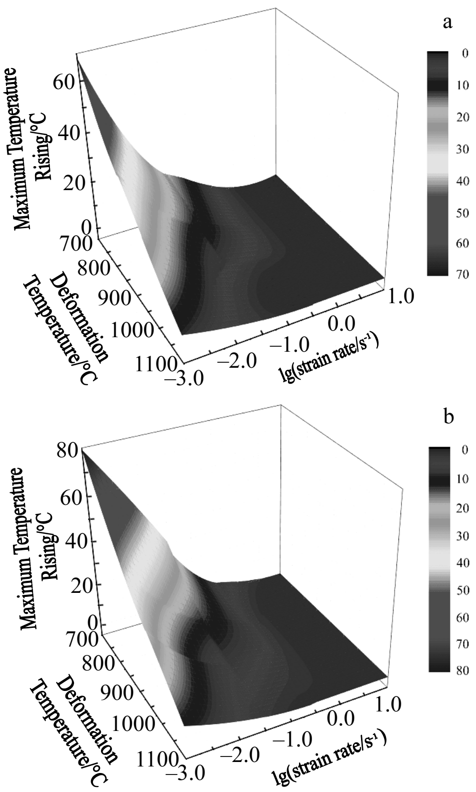

Fig.5 Three-dimension maps of the maximum temperature rising value of Ti-5553 alloys during hot deformation: (a) PM alloy and (b) IM alloy

process in a shorter time, which leads to a higher degree of ATR. As for the influence of temperature, the two alloys have lower deformation resistance (Fig.2) and more active softening mechanisms at relatively higher temperatures, reducing ATR subsequently.

Furthermore, PM alloy shows a lower degree of ATR effect than IM alloy under the same deformation condition. As shown in Fig.2, PM alloy shows lower peak stress and lower flow stress than IM alloy under the same processing condition, demonstrating the lower deformation resistance of PM alloy. Moreover, as revealed in Fig.1, IM alloy has larger grain size, which is ten times larger than the size of PM alloy. Not only that, IM alloy also has more precipitates than PM alloy before deformation. Viscous flow effect of grain boundaries is easier to be activated in the titanium alloys with finer grain and more β phase, by which the deformation compatibility can be improved significantly[20,21]. Lower deformation resistance and higher deformation compatibility make it easier for the external energy to be transformed into the internal energy of the alloy or to be dissipated by thermal radiation, and thus the degree of ATR for PM alloy is weaker than that for IM alloy. Furthermore, different softening mechanisms under various conditions of the two alloy may also contribute to different ATR degrees, which is worthy of further investigation.

3 Conclusions

1) The degree of DYP shows a positive correlation to strain rate but a negative correlation to deformation temperature for both PM and IM alloys, and the occurrence of DYP in the alloys is elucidated by the dynamic theory.

2) IM alloy shows a higher degree of DYP than PM alloy under the same processing condition, which Can be ascribed to IM alloy's low initial dislocation density in as-cast state and the subsequently promoted newly-generated mobile dislocation from grain boundary.

3) PM alloy shows a lower degree of ATR effect than IM alloy under the same deformation condition, because PM alloy has lower deformation resistance and higher deformation compatibility.

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粉末冶金态与铸态 Ti-5553 合金高温不连续屈服行为和绝热温升效应对比研究

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摘 要：研究了粉末冶金态与铸态 Ti-5553 合金在温度为 700~1100 ℃、应变速率为 0.001~10 s^{-1} 条件下的高温不连续屈服行为和绝热温升效应，并对这 2 种同名义成分不同制备工艺的钛合金进行了对比研究。结果表明：2 种合金不连续屈服的幅度均与应变速率呈正相关关系，并与温度呈近似负相关关系，2 种合金中出现的不连续屈服现象符合动态理论。在相同变形条件下，铸态合金中不连续屈服的幅度更大，其原因是相对于粉末冶金态合金，铸态合金中的起始位错密度低，这更有利于晶界处可动位错的突然增殖与扩展。2 种合金在热变形中绝热温升的大小均随应变速率的升高而逐渐增大，并随着变形温度的升高而逐渐降低。在相同变形条件下，粉末冶金态合金的绝热温升效应相比与铸态合金较弱，这是因为粉末冶金态合金具有较低的变形抗力和较高的协调变形能力。

关键词：Ti-5553 合金；粉末冶金；热变形；不连续屈服；绝热温升

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