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Deformability and microstructure transformation of PM TiAl alloy prepared by pseudo-HIP technology

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Abstract: Microstructures and deformation properties of Ti-46Al-(Cr, Nb, W, B) alloy consolidated by pseudo-HIP technology were investigated. The results show that the pseudo-HIP temperature has a significant effect on microstructures. When the sintering temperature is 1 100 ℃, the microstructure of as-pseudo-HIPped alloy is similar to that of the prealloyed powder and the interfaces of these powder particles are still discernible, but a near γ microstructure appears in particles. Increasing the pressing temperature to 1 200 ℃ develops successfully a homogeneous and fine-grained duplex microstructure. A typically fully lamellar microstructure with residual β phase is developed at 1 300 ℃. The compact exhibits excellent deformation properties at elevated temperatures. When the compression temperature is higher than 1 100 ℃, high quality products without cracks can be obtained even if the engineering compression strain is up to 0.8 at strain rates of 10^{-2}–10^{-3} s^{-1}. It can be established that the mechanical twinning and matrix deformation due to ordinary dislocation slip/climb contribute to the whole hot deformation.

Key words: titanium aluminides; pseudo-hot isostatic pressing; powder metallurgy; microstructure; deformability

1 Introduction

TiAl alloys have attracted much attention because of their potentially attractive properties such as low density, good modulus retention and high oxidation resistance[1−2]. A viable PM process[3−4] for producing high quality TiAl alloys is using atomized alloy powder produced by plasma rotating electrode process (PREP) and hot isostatic pressing (HIP). Small compositional variations can exert a significant effect on the strength and the ductility of TiAl based alloys, so the uniform composition obtained by PM processing is particularly attractive[5−6]. Consistent process and cost reductions also make PM TiAl an alternative high temperature material[7]. But, HIP is rather expensive due to the equipment involved, and new process such as pseudo-HIP process is developed[8].

The pseudo-HIP process (Fig.1) focuses on time reduction for consolidation. Atomized powder is filled in steel cans, sealed, preheated and degassed. The container is inserted in a pot die of a hydraulic press at elevated temperatures. The die is filled with an easily deformable and thermally stable powdered material of low thermal conductivity like talcum or pyrophillite which surrounds and supports the capsule. The granular material around the container is then pressurized by a piston. The pressure is transmitted to the can, leading to full density within a few minutes. The pressure vessel remains more or less cold because of the few cycling time and the insulation effect of the embedded material. The consolidated cans are subsequently hot worked for further densification. In the present study, the Ti-46Al-(Cr, Nb, W, B) alloy is prepared by pseudo-HIPping powder produced by PREP, and the microstructure and the deformation properties at elevated temperatures are examined.

2 Experimental

Ti-46Al-(Cr, Nb, W, B) (molar fraction, %) alloy powder was produced by PREP. The results showed that the powder had a uniform composition. The oxygen and nitrogen contents, measured by the inert gas melting-IR

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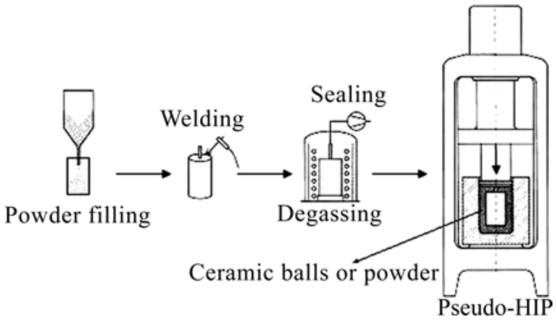

Fig.1 Schematic diagram for pseudo-HIP process

absorption spectrometry, were less than 6.8×10^{-4} and 3.2×10^{-5}, respectively.

Alloy powder with particle size of 100−150 μm was filled into steel can and degassed at 400 ℃. Alumina powder was used as the medium for pseudo isostatic pressure. The pressure of 140 MPa was applied to the pseudo-HIP chamber with a conventional unaxial pressing machine at temperature between 1 100 ℃ and 1 300 ℃. The pressure was transmitted to the can, leading to full density within 2 min. Density measurements were made on specimens coated with vaseline, using the Archimedean method. The relative density was calculated using the reference value of 3.9 g/cm^3.

Cylindrical samples with *d*10 mm×12 mm were cut by electric-discharge. To entrap the lubricant, the ends of the specimens were recessed to a depth of 0.2 mm. The high temperature compression tests were conducted on Gleeble−1500 thermo-simulation machine at the temperatures ranging from 1 100 ℃ to 1 200 ℃ and strain rates of 10^{-3}, 10^{-2} and 10^{-1} s^{-1}. Specimens were heated by induction coils with a heating rate of 10 ℃/s and soaked for 5 min at the test temperature before compression tests. Optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used for the examination of the morphology and microstructures of the powder, the compact and the deformed specimens.

3 Results

3.1 Characteristics of Ti-Al-Cr-Nb-W-B alloy powder and compact

The average particle size of as-received powder is approximately 125 μm. The particles are of spherical shape with inhomogeneous size, as shown in Fig.2(a). Fine particles were sieved and used for the experiments. Most of the particles have a dendrite surface morphology. The backscattered electron image of the particle cross-section (Fig.2(b)) shows a rapidly-solidified cellular dendritic structure. The cell size varies between 5 μm and 20 μm. Typically, as the atomized powder solidifies,

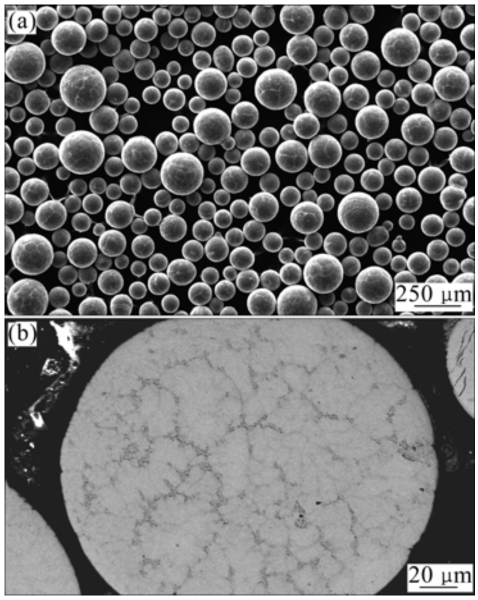

Fig.2 Morphologies of dendritic surface (a) and cross-section (b) of Ti-46Al-(Cr, Nb, W, B) alloy powder

α phase is the first phase to form from the undercooled liquid. So, the interdendritic regions solidify by peritectic reaction to form *α* phase.

The appearance of pseudo-HIPped compact is shown in Fig.3. The compact has a regular shape without internal and external cracks, and no oxidization scale is found. A relative density of >99.5% is achieved in the as-pseudo-HIPped compacts at pressing temperature higher than 1 100 ℃.

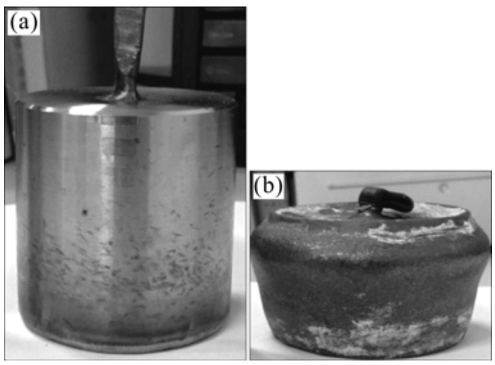

Fig.3 Appearance of Ti-46Al-(Cr, Nb, W, B) alloy compact fabricated by pseudo-HIP technology: (a) Before pressing; (b) After pressing

The microstructures formed during pseudo-HIP process are found to dramatically change with the increase of pressing temperature, as shown in Fig.4. The alloying powders are deformed and bonded, but the interfaces of these particles are very evident at 1 100 ℃ (Fig.4(a)). The compact has a dendritic microstructure, similar to that of the prealloyed powder (Fig.4(b)). When

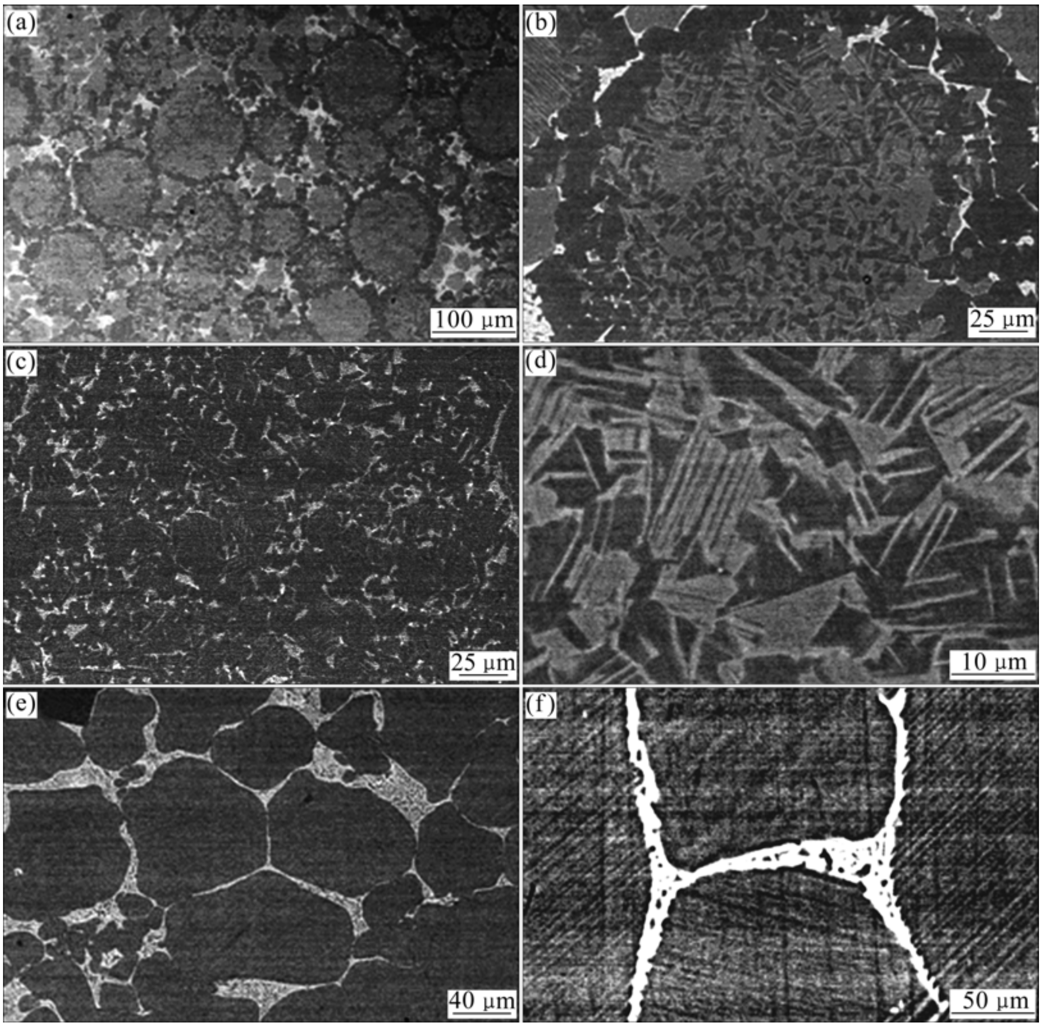

Fig.4 Microstructures of Ti-46Al-(Cr, Nb, W, B) alloy pseudo-HIPped at different temperatures: (a), (b) 1 100 ℃; (c), (d) 1 200 ℃; (e), (f) 1 300 ℃

the pressing temperature is up to 1 200 ℃, a duplex microstructure with a grain size of 10 μm appears (Fig.4(c)). It also contains a small amount of β phase, appearing white in the BSE image (Fig.4(d)). A typically fully lamellar microstructure containing fine and homogeneous lamellar colonies α_2/γ is developed at 1 300 ℃. The average lamellar colony size is about 100 μm (Fig.4(e)). In addition, it can be seen that there are composition segregations in the fully lamellar microstructure from the BSE micrographs (Fig.4(f)). Therefore, it can be deduced that the pressing temperature has a significant effect on microstructures, and 1 200 ℃ is an appropriate pressing temperature to fabricate TiAl alloys with duplex microstructure, consisting of fine and homogeneous γ equiaxed grains and α_2/γ lamellar colonies.

3.2 Deformation properties

The hot compression experiments were carried out from 1 000 to 1 200 ℃ at three strain rates. The compression properties at elevated temperatures for Ti-46Al-(Cr, Nb, W, B) alloy are shown in Fig.5.

For each sample, the flow stress decreases and the

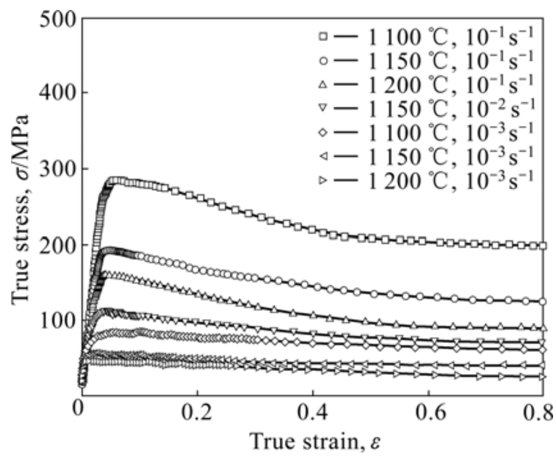

Fig.5 True stress and true strain curves at various temperatures during hot compression of as-pseudo-HIPped Ti-46Al-(Cr, Nb, W, B) alloys

shape of the stress—strain curves changes with the increase of compression temperature. The shapes of the stress—strain curves in high strain rates of 10^{-1} and 10^{-2} s^{-1} exhibit the similar trends, having a characteristic maximum at the initial stage of the deformation. But at low strain rate of 10^{-3} s^{-1}, the stress—strain curves become essentially flat (parallel to the horizontal axis) and exhibit no work hardening after yielding, showing that the alloy has a good hot workability[9].

3.3 Microstructure evolution during deformation

The microstructures of the central sections of compression samples with true strain of 0.8 in different deformation conditions are shown in Fig.6. The results show that the microstructure of as-pseudo-HIPped samples has a similar structure with fine γ equiaxed grains (<5 μm), and the grain size is refined obviously. Such a structure resembles to the structures of the alloys exhibiting superplasticity at elevated temperatures.

4 Discussion

4.1 Effect of pressing temperature on microstructures

During pseudo-HIP process, the initial structures of alloy powders disappear with increasing the pressing temperature. A small amount of α_2 phase forms at 1 100 ℃, which is related to the transformation of $\alpha\rightarrow\gamma+\alpha_2$. With increasing the pressing temperature to 1 200 ℃, the microstructure is essentially the duplex structure consisting of fine and homogeneous γ equiaxed grains and α_2/γ lamellar colonies, but still having residual β phase. Higher pressing temperatures increase the content of lamellar colonies at the expense of γ equiaxed grains and coarsening of the microstructures.

When the temperature increases to 1 300 ℃, a slightly coarser fully lamellar microstructure forms in terms of a strict orientation relationship, but the lamellar colony size is relatively small. The lamellar microstructure is probably defined by: $\alpha\rightarrow\alpha+\gamma\rightarrow\gamma+\alpha_2\rightarrow$ lamellar(α_2/γ) transformation[10]. The formation of α_2/γ lamellar structures in the as-pseudo-HIPped alloys is different from that in ingot. Generally, it is a solidification process for the ingot. However, the microstructural evolution in the as-Pseudo-HIPped alloys is a diffusion-controlled phase transformation[10−11]. During solidification of powder particles, β phase as the first solidified phase becomes rich in Cr, Nb and W. However, the volume fraction of β phase is small due to the lower concentrations of Cr, Nb and W in the interdendritic region. Subsequently, β phase transforms to α. However, the process is incomplete because the cooling rate is too rapid to completely eliminate differences in composition by diffusion. During further cooling, very fine β phase remains and locates in the lamellar colonies. Lamellar colonies and β phase may simultaneously grow up, but the growth rate of lamellar

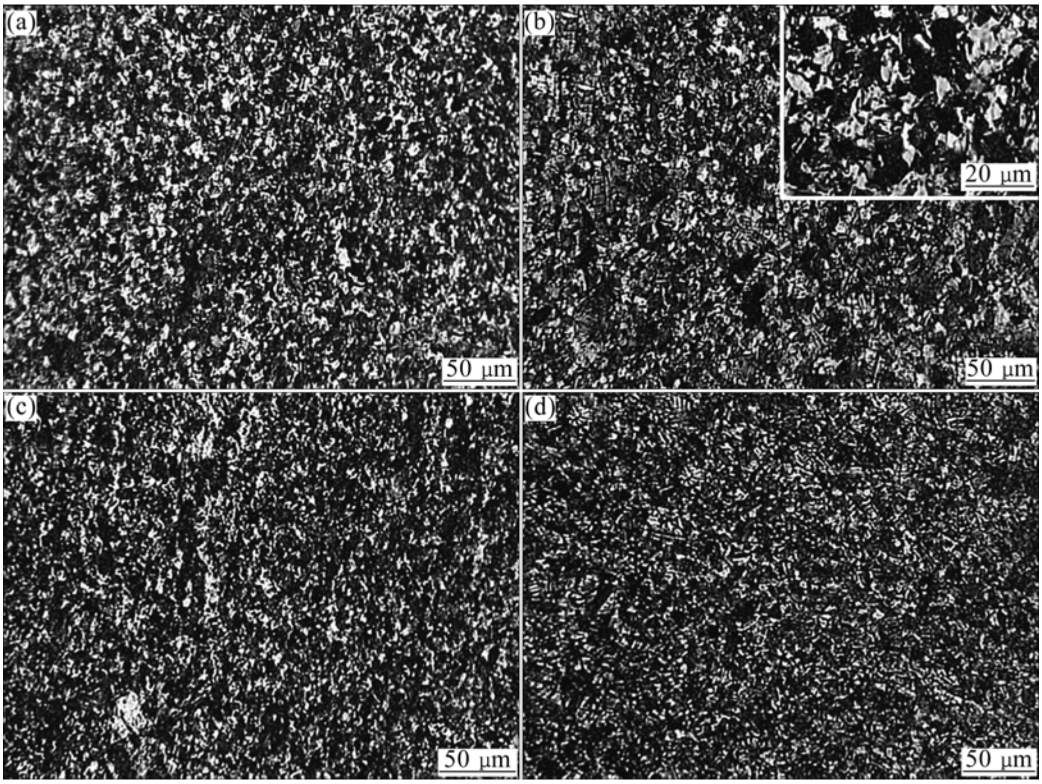

Fig.6 Microstructures of PM Ti-46Al-(Cr, Nb, W, B) alloy in different deformation conditions and at true strain of 0.8: (a) 1 000 ℃, 10^{-3} s^{-1}; (b) 1 100 ℃, 10^{-3} s^{-1}; (c) 1 200 ℃, 10^{-1} s^{-1}; (d) 1 200 ℃, 10^{-2} s^{-1}

colonies is faster than that of β phase due to the low diffusivities of Cr, Nb and W.

4.2 Deformation mechanism of alloy

To better understand the flow behaviors in hot deformation, the stress exponent n and the activation energy Q were obtained by the following methods.

From these experimental results, it can be established that the effect of the strain rate on deformation behavior is significant. With the increase of the strain rate, the compression temperature increases if high quality compressed samples without cracks can be obtained. It is well known that the compression deformation is complied with Zener–Hollomon parameter Z, i.e.

$$Z=\dot{\varepsilon}\exp[Q/RT] \tag{1}$$

where $\dot{\varepsilon}$ and T are the test strain rate and temperature, respectively; and Q is the apparent activation energy for hot deformation. Using the kinetic rate equation, the stress exponent n and the apparent activation energy Q are determined to be 2.48 and 319 kJ/mol, respectively. It is indicated that the alloy has a lower deformation resistance at high temperatures and low strain rates.

TEM observation reveals that many deformation twinning and intergranular dislocations are found in various deformation conditions (Fig.7). In the early stages of deformation, the flow behavior is mainly governed by twinning and dislocation slip. Figs.7(a) and (b) show twins in deformed microstructure after compression at 1 000 ℃. In TiAl only, four twinning systems are active because the other systems would destroy the ordered structure of TiAl[12]. These unidirectionally operating twinning systems cause an asymmetry of the yield surfaces polyhedron of the slip systems of ordinary dislocations, resulting in a certain orientation relationship[13−14].

After adequate amounts of deformation, the dislocation slip/climb and mechanical twinning offer conditions necessary for dynamic recovery and dynamic recrystallization (DRX)[15] (Fig.8). The dislocation climb process (Fig.7(c)) frequently accompanies grain matrix deformation because many dislocations in pile-ups at grain boundaries can climb easily at high temperatures, thereby relieving the stress concentration. The ductility of alloy can thus be improved due to the extensive dynamic recovery, and correspondingly, the true stress—strain curves exhibit a flow softening behavior as shown in Fig.5. On the other hand, the small starting grain size of the PM samples may improve the DRX reaction kinetics.

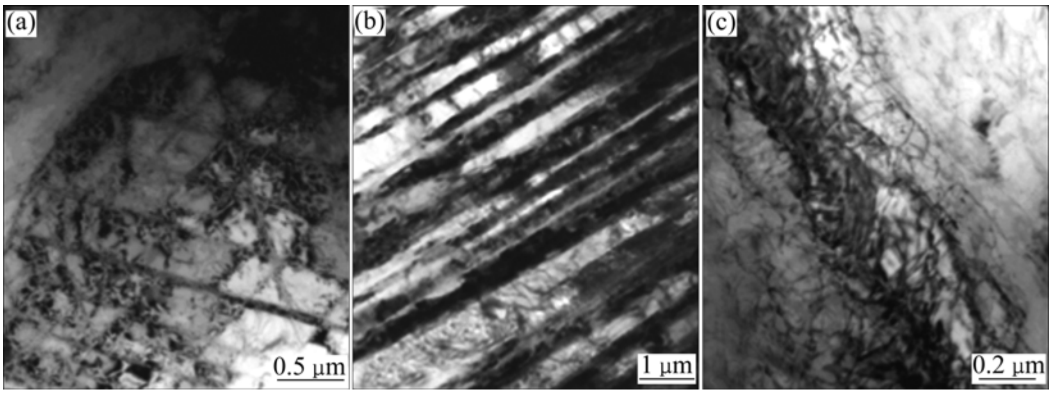

Fig.7 Typical microstructures of Ti-46Al-(Cr, Nb, W, B) alloy after hot compression tests: (a) and (b) Twin grains; (c) Interfacial dislocation array

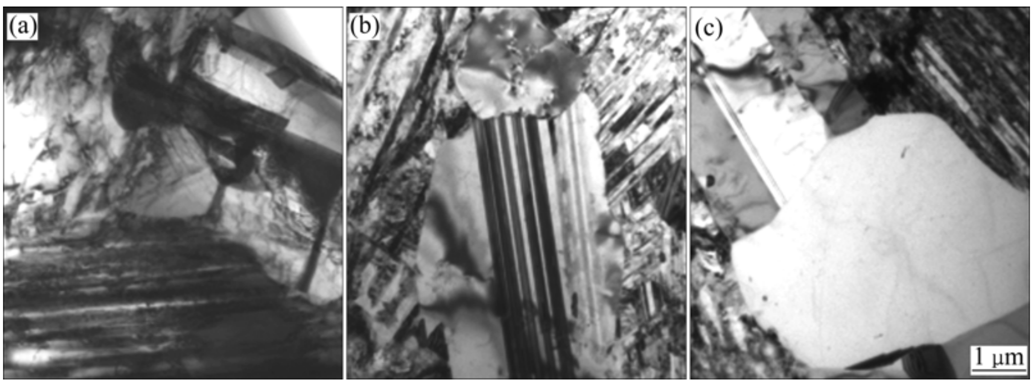

Fig.8 TEM images showing typical DRX grains of Ti-46Al-(Cr, Nb, W, B) alloy after hot compression tests

5 Conclusions

1) The pseudo-HIP temperature has a significant effect on the microstructures of Ti-46Al-(Cr, Nb, W, B) alloy. When the sintering temperature is 1 100 ℃, the microstructure of the as-pseudo-HIPped alloy is similar to that of the prealloyed powder, and the interfaces of these powder particles are still discernible, but a near γ microstructure appears in the particles. Increasing the pressing temperature to 1 200 ℃ develops successfully a homogeneous and fine-grained duplex microstructure. A typically fully lamellar microstructure with residual β phase is developed at 1 300 ℃.

2) The compact exhibits excellent deformation properties at elevated temperatures. When the compression temperature is higher than 1 100 ℃, high quality compressed samples without cracks can be obtained even if an engineering compression strain is up to 0.8 at the strain rates from 1×10^{-2} to 10^{-3} s^{-1}. It can be established that mechanical twinning and matrix deformation due to ordinary dislocation slip/climb contribute to the whole hot deformation process.

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