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MECHANICAL PROPERTIES OF POWDER TITANIUM AT DIFFERENT PRODUCTION STAGES. IV. MECHANICAL PROPERTIES AND CONTACT FORMATION IN POWDER TITANIUM PRODUCED BY DYNAMIC HOT PRESSING

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The paper examines the effect of hot pressing parameters on the structure and properties of powder titanium. The initial porosity of the compact, sintering temperature and time, and temperature of dynamic hot pressing are varied. The resistivity and mechanical properties are used to assess the quality of the compacts after pressing. It is shown that powder titanium produced under optimal conditions compares well with commercially pure titanium produced with a conventional method.

Keywords: *porosity, powder titanium, hot pressing, elastic modulus, conductivity, bending and tensile strength.*

INTRODUCTION

Conventional cold pressing and sintering of powder titanium produce compacts with low porosity (~5%) and yield stress and elastic modulus comparable with the properties of compact titanium [1]. However, such parts have low plasticity and, thus, low fracture toughness and crack resistance. In addition, to avoid interparticle fracture, sintering should be conducted at very high temperatures (1100–1200°C), which may adversely affect the structure and phase composition of titanium alloys. Hot pressing decreases the porosity of compacts made of plastic powders to almost zero and promotes perfect contact at much lower temperatures than conventional pressing and sintering do [2].

We examined how contact forms in titanium when welded under pressure [3] and found out that perfect contact formed in 20 min at a temperature of 650 to 700°C and a strain of about 20% in the contact area. This testifies that hot pressing is promising for making titanium parts. Cold welding is conducted in vacuum since rutile that forms over a part when heated in air makes contact extremely difficult to form. This is also the case for powder titanium parts: either oxygen access should be prevented when they are heated or high-temperature compaction should be conducted in vacuum. In the latter case, the effectiveness depends on whether all production operations can be automated and whether the process time can be drastically reduced, up to high strain rates.

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The objective of this paper is to examine contact formation in powder titanium produced by dynamic hot pressing (DHP).

EXPERIMENTAL PROCEDURE

We used PTÉS titanium powder, which was sieved into –063, –063+0.5, and –0315+0.2 fractions. Cylindrical billets 25 mm in diameter and 10 mm in height were produced by cold pressing in 10 and 20 t presses. Their porosity was 30 and 20%, respectively. Hot pressing was conducted using an experimental vacuum unit developed at the Institute for Problems of Materials Science (National Academy of Sciences of Ukraine) [4]. Samples for dynamic hot pressing were heated for 10–15 min and then held at the pressing temperature for 20 min. Hot pressing was conducted at 20, 300, 600, 800, and 950°C for about $3 \cdot 10^{-3}$ sec. The residual porosity and electrical and mechanical properties of the parts were examined. The quality of contacts under different deformation conditions was studied using the methods described in [1]. The fracture patterns were analyzed with a Superprobe-723 microscope.

EXPERIMENTAL RESULTS

Table 1 summarizes data on the effect of hot pressing temperature on the porosity and electrical properties of samples made of –0315+02 powder titanium and data on the effect of sintering temperature on the properties of cold-pressed compacts made of powder of the same fraction. Dynamic pressing densifies the compacts to 1.8% porosity at room temperature and to 0.8% porosity at 300°C, the compacts becoming porousless at higher temperatures (starting from 600°C).

The resistivity of DHP samples produced at room temperature was 1083 $\Omega \cdot$ m. This value is not only one order of magnitude higher than that of compact titanium ($45 \cdot 10^{-8}\ \Omega \cdot$ m [5]), but also twice that of green titanium compacts with a porosity of 5%, which seems to be associated with the strong elastic aftereffect during the unloading of the compact after dynamic pressing. The resistivity drops after increase in DHP temperature. For example, after pressing at 300°C, the resistivity abruptly decreases (to 145 $\Omega \cdot$ m) and approaches that of compact titanium. Note that sintering of pressed compacts at 300°C insignificantly changes their resistivity as compared with green compacts. After DHP at higher temperatures, the resistivity of the compact is closely similar to that of compact titanium.

Because of the small sizes of the samples, the elastic modulus could not be measured with the methods described in [6]. Hence, the quality of interparticle contact was assessed using the yield stress, which changes with

TABLE 1. Effect of Hot Pressing and Sintering Temperatures on the Electrical Properties of –0315+02 Powder Titanium Samples

Temperature, °C	Porosity, %	Resistivity, $\Omega \cdot$ m, 10^{-8}	Conductivity, $1/\Omega \cdot$ m, 10^{8}
DHP			
20	1.8	1083.0	0.00092
300	0.8	145.0	0.0066
600	0	47.5	0.021
800	0	50.0	0.02
950	0	50.0	0.02
Sintering			
20	5	630.0	0.0018
300	5	450.5	0.0023
500	5	120.0	0.0081
700	5	62.5	0.0182
1000	5	48.9	0.021
1200	5	48.9	0.021

TABLE 2. Effect of Hot Pressing and Sintering Temperatures on the Mechanical Properties of–0315+02 Powder Titanium Samples

T, °C	Test*	E, GPa	σ_{001}, MPa	σ_{02}, MPa	σ_{max}, MPa	e_p
DHP						
25	Bending	7.7	12.3	17.3	20.0	0.0045
300	“	19.5	47.3	67.1	68.5	0.0020
600	“	–	232.7	341.3	366.0	0.0043
800	Tension	–	271.0	325.0	370.0	0.4200
950	“	–	283.0	347.0	435.0	0.7000
Sintering						
25	Bending	9.83	5.7	17.5	20.7	0.0048
300	“	10.1	5.2	15.7	18.6	0.0035
500	“	11.3	11.2	19.4	19.6	0.0022
700	“	42.0	30.7	76.7	80.7	0.0027
1000	Tension	92.0	–	250.0	250.0	0.0710
1200	“	95.0	–	300.0	350.0	0.3200

* The values of bending yield stress were divided by 1.5 to be compared with uniaxial tension [8].

sintering temperature in practically the same manner as the elastic modulus does [7]. Table 2 summarizes the mechanical properties of the samples. The yield stress abruptly increases with hot pressing temperature. After DHP at room temperature, the billet has the yield stress comparable with that of dense green compacts produced with a conventional method. The yield stress of compacts pressed at 300°C substantially increases but remains much less than that of compact, commercially pure titanium ($\sigma_{02} \sim 300$ MPa) [3]. The yield stress of the DHP samples produced at 600°C reaches almost the same level, but their fracture ductility e_r remains very low.

After DHP at 800°C, the ductility of samples increases to an extent that they bend by more than 90° without failure in bending tests. Therefore, these samples, as well as the samples pressed at 950°C, were tested by uniaxial tension. The samples produced by DHP at 800 and 950°C show quite high uniform strain (to 10%), followed by necking. In the case of tension, the rupture strain was determined as the logarithm of the initial cross-sectional area S_i of the sample divided by the cross-sectional area S_f of the neck at failure:

$$e_r = \ln(S_i / S_f). \tag{1}$$

The samples produced by DHP at 800°C have the yield stress comparable with that of recrystallized titanium, and those produced at 950°C have somewhat higher yield stress (Table 2). The fracture ductility of the samples pressed at 800°C varies between 0.3 and 0.5, which is approximately 100 times higher than that of the samples pressed at lower temperature but is about one-third that of commercially pure titanium produced by a conventional method. Dynamic hot pressing at 950°C permits ductility $e_r = 0.65$–0.75, which is only insignificantly (by 20%) lower than that of VT 1.0 recrystallized titanium [3].

For better insight into the advantages and disadvantages of DHP, we compared the quality of contacts in hot-pressed and sintered samples. For comparison, we determined coefficients to characterize the quality of electrical (K_λ), mechanical (K_E or $K_{\sigma_{02}}$), and physical (perfect) (K_e) contacts using the method described in [1]. The coefficients were calculated as follows:

$$K_\lambda = [(\lambda_{ms} - \lambda_{gr})] / [(\lambda_{th} - \lambda_{gr})] \cdot 100\%$$

for electrical contact,

$$K_{\sigma_{02}} = [(\sigma_{ms} - \sigma_{gr})] / [(\sigma_{th} - \sigma_{gr})] \cdot 100\%$$

for mechanical contact,

$$K_{e_p} = [(e_{ms} - e_{r_{gr}})] / [(e_{r_{th}} - e_{r_{gr}})] \cdot 100\%,$$

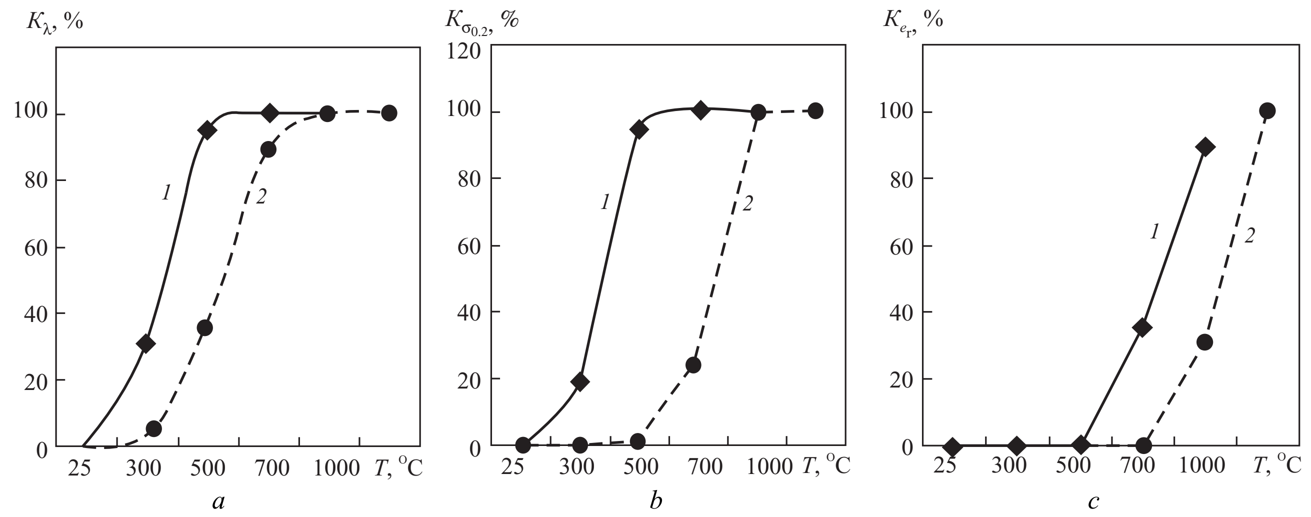


Fig. 1. Temperature dependence of coefficients that characterize the quality of electrical (*a*), mechanical (*b*), and physical (*c*) contacts of samples produced by DHP (*1*) and sintering (*2*)

and for physical contact (here λ, σ, and e_r are conductivity, yield stress, and rupture strain; the subscript 'th' refers to perfect contact, 'gr' to a green sample, and 'ms' to a sample produced under preset thermomechanical conditions). The results are summarized in Table 3.

Based on Table 3, temperature dependences of the quality of contacts in samples produced by DHP and sintering were plotted (Fig. 1). It is seen that mechanical, electrical, and physical contacts form in DHP well before they do in sintering. For example, the contacts are 50% complete at the following temperatures: electrical contact at 375°C in DHP and at 585°C in sintering, mechanical contact at 450 and 730°C, and physical contact at 860 and 1120°C. These differences are because deformation defects interact with the contacting surfaces in DHP and transfer some part of their energy to them, thus intensifying the contact formation. In sintering, deformation defects just accelerate the diffusion of atoms into the contact area.

As with sintering, contact formation depends on the hot-pressing time. The role of kinetic factor may be ascertained in comparing DHP results with data obtained in titanium welding under pressure [3], when contacts form in vacuum under the action of temperature and deformation. Cold welding lasted for 20 min, with the strain in the contact areas being 20%. [3]. Table 4 summarizes the results.

TABLE 3. Effect of DHP and Sintering Temperature on the Coefficients That Characterize the Contacts in –0315+02 Powder Titanium Samples

T, °C	K_λ, %	$K_{\sigma_{02}}$, %	K_{e_r}, %
		DHP	
25	0	0	0
300	31	19	0
600	100	100	0
800	100	100	35
950	100	100	75
		Sintering	
25	0	5	0
300	3	0	0
500	36	1	0
700	89	26	0
1000	100	100	23
1200	100	100	100

The mechanical contact is clearly observed at the minimum welding temperature of 600°C (the yield stress is comparable with that of compact titanium). The physical contact is absent at this temperature: rupture strain of this sample is very low ($e_r = 0.018$), and the seam fails along contacting planes. In the process, absolutely smooth surfaces suffered fracture. At 650°C, contact takes on mechanical properties, which are at least not worse than those of the base metal, and the sample fails by necking beyond the seam. A metallographic analysis (Fig. 2) showed recrystallization at the interface near the seam and a new grain formed at the contact area, which combines the parts of the sample being welded.

Tables 2 and 4 show that the samples produced by DHP and cold welding under the optimal deformation conditions have close mechanical properties. Only do temperature and rate at which high-quality contact forms differ, temperature being 200–300°C higher in DHP. This difference may be attributed to the kinetics of contact formation: contacts form very quickly in DHP ($\sim 3 \cdot 10^{-3}$ sec).

To analyze the physical factors responsible for contact structurization in DHP, structural rearrangement during hot pressing should be examined. As noted in [1], the formation of mechanical contact is associated with healing of interparticle two-dimensional pores that originate when plastic powder is compacted. A fracture analysis

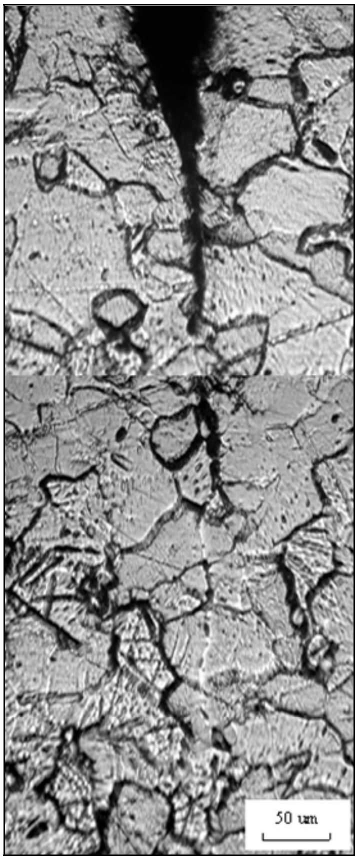
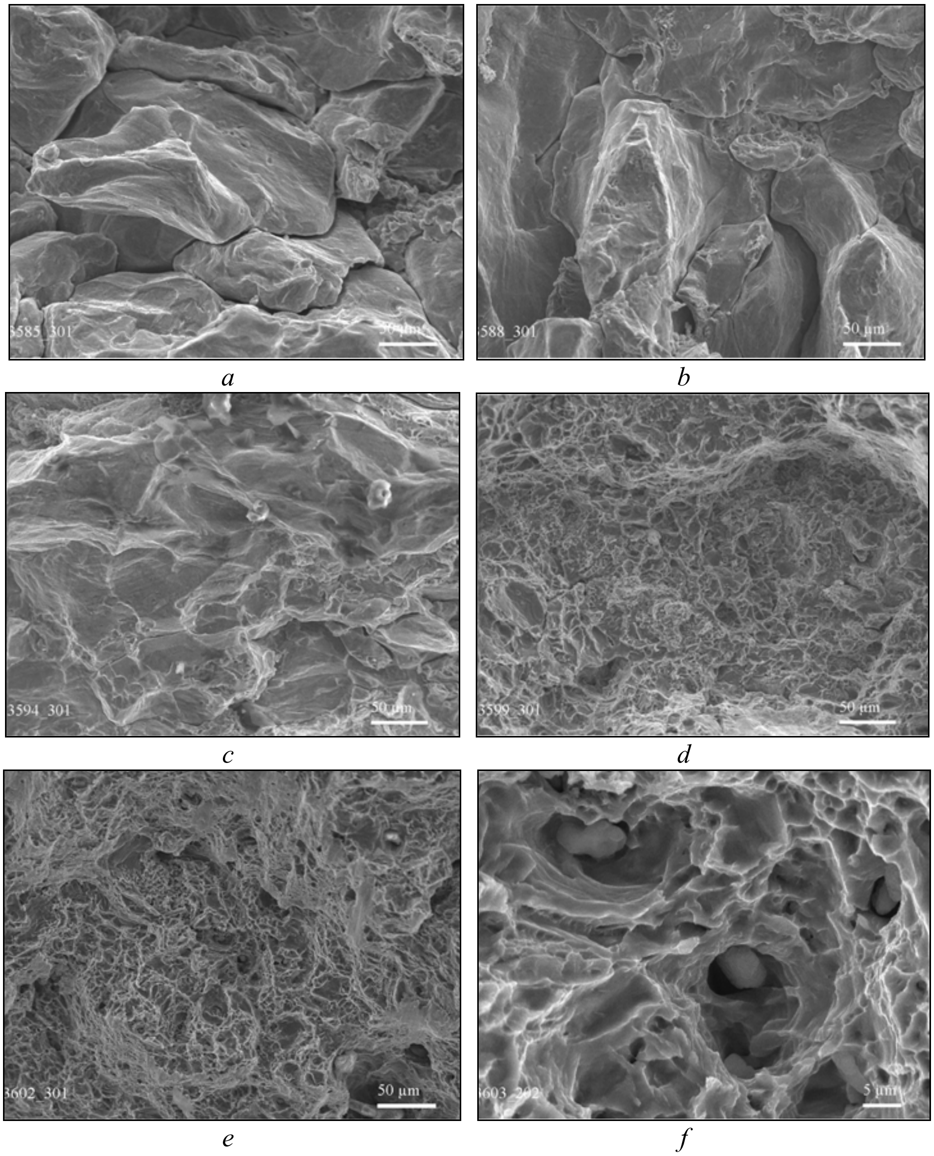


Fig. 2. Microstructure of a sample after cold welding at $T = 700$°C

TABLE 4. Research Results for Titanium Samples Subjected to Cold Welding under Pressure [3]

Treatment	T_{weld}, °C	e_{fr}	ψ, %	ε_{eq}, %	σ_y, MPa	σ_b, MPa	σ_v, MPa	Failure spot
Initial	20	0.82	61	10	350	463	830	Sample
Welding	600	0.02	2	2	273	348	350	Seam
”	650	0.78	62	7.6	340	420	815	Sample
”	700	0.91	63	7.5	310	405	750	”



a *b* *c* *d* *e* *f*

Fig. 3. Fracture patterns of samples produced by DHP at 20 (*a*), 300 (*b*), 600 (*c*), 800 (*d*), and 950°C (*e*, *f*)

is an efficient method of structural research in this case, which shows the structural evolution at the contact area after DHP at different temperatures.

Figure 3 summarizes the results of fracture analysis. After dynamic pressing at room temperature, there are two-dimensional pores about 3 μm wide on the fracture area of the sample (Fig. 3a). After DHP at 300°C, areas of mechanical contact between particles are observed along with interparticle pores (Fig. 3b); after DHP at 600°C, there are practically no two-dimensional pores though fracture still occurs along interparticle boundaries (Fig. 3c). With further increase in the DHP temperature to 800°C, the fracture becomes mixed: fragments of interparticle fracture alternate with dimple fracture areas (Fig. 3d). Finally, dimple fracture predominates at 950°C (Fig. 3e, f).

Comparing the data from the fracture analysis and temperature dependences of the coefficients that characterize contact quality shows that the electrical and mechanical contacts form when interparticle cracks, which are seen in the fracture patterns of green samples, are healed. The physical contact forms when large-angle interparticle boundaries are recrystallized (Fig. 2). The recrystallization of interparticle boundaries leads to intraparticle dimple fracture over the entire contact area. In this case, the mechanical properties of the DHP samples are closely similar to those of conventionally produced titanium.

CONCLUSIONS

Electrical, mechanical, and physical contacts form in powder titanium in DHP well before they do in sintering. The 50% electrical contact forms at 375°C after DHP and at 585°C after sintering, the 50% mechanical contact at 450 and 730°C, and the 50% physical contact at 860 and 1120°C, respectively.

The physical contact forms at much higher temperature (by 200°C) in DHP than in cold welding but in a much shorter time.

The optimal DHP conditions are T_{def} = 950°C and 20% porosity of the starting compact. The properties of such powder titanium compacts are as good as those of commercially pure titanium produced with conventional methods.

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