DYNAMIC COMPRESSION OF TITANIUM MONONICKELIDE

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In this work, the behavior of powdered titanium mononickelide in static and shock compression and subsequent heating of the molds was studied using the methods of x-ray and dilatometric analysis. It was shown that during static compression, in distinction from shock compression, the phase transition B2 → B19 takes place to a significant degree in the powder. At the same time, shock compression causes significantly more lattice defects in comparison with static compression, which the process of subsequent sintering activates. During the sintering of the pressings produced by static compression, the reverse transformation takes place in them accompanied by the effect of shape "memory" and causing the distension of the molds, which complicates the production of high-density sintered material.

The intermetallic compound titanium mononickelide (TiNi), due to its unique properties (shape "memory," mechanical load damping, superelasticity) can be used quite effectively in different branches of technology. In connection with this, the production of manufactured goods of TiNi and of compositions containing this material using the methods of powder metallurgy is of interest. However, the properties of TiNi mentioned above, which can be useful for using manufactured goods of such compositions, complicate their preparation using the traditional methods of powder metallurgy. In the first place, this pertains to the effect of shape "memory." This effect is caused by the occurrence of the inverse martensite transformation in TiNi when changing the temperature or during mechanical loading. The temperature interval of the transformation is found in the area of room temperature and can be changed with a small change in the chemical composition of TiNi [1]. Thus, the compression of a powder which is usually done at room temperature can cause the occurrence of the martensite transformation of the high-temperature phase having a B2 type ordered bcc-lattice into the low-temperature phase having lower symmetry. It is thought that in pure titanium mononickelide this phase has a type B19' lattice (type B19 with monoclinic distortion). The formation of this phase is possible having small triclinic distortion – B19" [2]. When heating the pressure during subsequent sintering, the inverse transformation can be expected accompanied by the effect of shape "memory," which can cause distension of the pressing in the sintering process and thereby inhibit the production of a high-density manufactured article.

Another important question when producing sintered articles from compositions containing titanium nickelide is the problem of interphase interaction of TiNi, for example, with refractory compounds when sintering compositions of the hard alloy variety. In the course of liquid phase sintering of similar compositions, undesirable processes take place which degrade the characteristics of the sintered products [3]. Therefore, such compression technologies seem promising which would activate the sintering process, lowering its temperature interval toward the region where only the solid phase exists.

In light of the aforementioned, there is great interest in the process of shock compression of compositions with a TiNi-binder. This process leads any crystal into a strongly excited state [4], which should inhibit the formation of low-temperature martensite during compression and permit sintering at decreased temperatures in the absence of a liquid phase. This work is devoted to the study of this process.

PN55T45 grade powder having a dispersion of up to 40 μm was used for the study. Before compression, the powder was annealed in a vacuum oven at 850°C for 1 h. Static compression of samples 15 mm in diameter and 10 mm high was done in a hydraulic press using the method of double-sided compression at pressures of up to 2000 MPa. The formation of the low-

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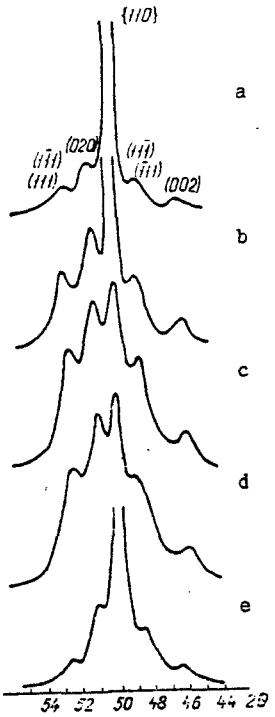

Fig. 1

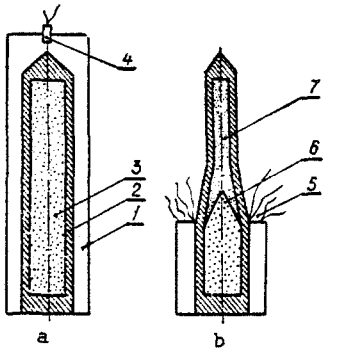

Fig. 2

temperature phases in the process of static compression of TiNi powder was confirmed by the results of investigating the phase composition of freely saturated and supersaturated powder at various pressures. The phase composition was studied on a DRON-2.0 devices in CoK_α- radiation. Figures 1a-d show diffraction patterns of a freely saturated solution and pressings produced at compression pressures of 300, 1000, and 2000 MPa. From the figure it is clear that in a freely saturated solution of annealed powder, the number of low-temperature phases is insignificant. In the pressings, the number of B19 phases increases with increasing compression pressure, reaching saturation at a pressure of about 1000 MPa. At this compression pressure, this phase becomes the dominant one in the pressings. In addition, a noticeable splitting of the (111) reflection evidently indicates a B19' → B19" transformation [2].

To study the shock wave compression of TiNi powder using the energy of an explosion, in this work we used one of the most widely used methods of shock compression [5]. Referring to Fig. 2, the powder 3 was placed in a cylindrical ampul 2 that was closed by plugs at both ends. The loading was accomplished by detonating a charge of explosive material 1, which was applied in a uniform layer around the ampul (Fig. 2a). The charge was triggered by detonator 4 such that a ring-shaped detonation wave slides along the surface of the ampul and generates a conical shock wave 6 in the powder (Fig. 2b). The merit of this method is in the relative simplicity of its realization, and the capability for compressing large volumes of material. The basic difficulty in applying this method consists in selecting the optimum loading parameters which would ensure preserving the ampul from destruction after shock wave transit and maintaining the uniformity of density and structure throughout the volume of compressed powder 7. The results of compression using shock wave energy depend on the kind and amount of explosive, the charge configuration, the properties of the ampul material and its wall thickness, and the thermophysical properties of the material being compressed. Samples of TiNi powder were studied in this work that were produced by shock compression in ampuls of grade 3 steel that had a 15 mm inside diameter, 3.5 mm wall thickness, and 180 mm height. A mixture of hexogen with sodium chloride in a 1:1 ratio was used as the explosive. The compression of the TiNi powder in these conditions ensures a uniform density throughout the volume of 13-16%.

Figure 1d shows a diffraction pattern of TiNi powder after explosive compression. Comparing it with previous diffraction patterns shows that the volume share of the low-temperature phase in the process of shock compression actually increases significantly less than in static compression.

For quantitative evaluation of the effect of the form of compression on the phase compression, the ratio of high and low temperature phases in the pressings was calculated

TABLE 1

Compression method and pressure	Vol. share of (B19' + B19"), %	(110) line broadening, deg
Freely saturated	26	0.55
Static, 300 MPa	41	0.70
Static, 1000 MPa	64	1.00
Static, 2000 MPa	64	1.00
Shock	30	1.20

from the diffraction patterns. If the B2 cell is chosen, as described in [6], then the reflections of the {111} planes of the B2 phases are transformed during martensite transformation into reflections of the B19 phases in the following manner:

$$(011) \rightarrow (020), \quad (01\bar{1}) \rightarrow (002), \quad (101) \rightarrow (111),$$
$$(\bar{1}01) \rightarrow (1\bar{1}1), \quad (110) \rightarrow (\bar{1}11), \quad (1\bar{1}0) \rightarrow (11\bar{1}).$$

Calling to attention the small dilatometric effect of the martensite transformations and the identical chemical composition of the B2 and B19 phases, the volume share η of the B19 phase can be determined in the following way:

$$\eta = I_{B19}/I_{B19} + I_{B2},$$

where I_{B_2} is the intensity of the {110} reflection of the B2 phases and I_{B19} is the total intensity of the (020), (002), (111), ($\bar{1}$11), (1$\bar{1}$1), and (11$\bar{1}$) reflections of the B19 phase.

The intensity of the reflections was determined by the area under the corresponding peaks on the diffraction patterns. The results of calculating the volume share of the low-temperature phases (B19' + B19") are given in Table 1.

Analysis of the diffraction patterns (judging by the {110} line width of the B2 phase) shows that compaction during shock compression is accompanied by significantly larger increase in lattice defects than in static compression. The results of evaluating widths of the {110} line are given in Table 1. The half-width of the (311) line, measured for a more accurate estimate of this effect of the form of compression, turned out to be equal to 3.2° for shock compression and 2.7° for static compression at 2000 MPa.

In addition to x-ray analysis for studying the level of occurrence of the martensite transformations as a function of the means and level of loading during compression, for estimating the temperature interval of the transformation, and also for determining the influence of the reverse martensite transformation during heating on the distension of the pressing, dilatometric analysis was done on TiNi samples that were compressed statically and by shock loading. The dilatometric curves of samples produced by static and shock compression are shown in Fig. 3. It is clear from these curves that the samples expand when heated up to temperatures around 500°C, i.e., until the processes causing shrinkage (plastic flow, diffusion processes, etc.) begin to a significant degree. However, if the effective coefficient of thermal expansion of samples produced by shock compression, calculated from the dilatogram, equals $1.3 \cdot 10^{-5}$ deg^{-1}, i.e., roughly equal to the room temperature coefficient of expansion of TiNi ($1.4 \cdot 10^{-5}$ deg^{-1}) [1], then the expansion of samples produced by static compression is roughly an order of magnitude greater: a sample 5 mm long is lengthened by 0.3 mm when heated to 500°C at a rate of 10° per minute (Fig. 3, curve a). The greatest degree of length increase of the sample is observed in the 30-300°C temperature interval in which the linear expansion is almost independent of compression pressure in the 300-2000 MPa range. This substantiates the proposition regarding the capacity for distension of pressings made of a powdered composition prepared by static compression in the initial stage of sintering. At the same time in pressings produced by shock compression, the distension during sintering does not take place (Fig. 3, curve b), which permits, using shock compression, the production of denser products of the indicated composition after sintering. So, if static compression of compositions containing 50% (by weight) TiC in 50% TiNi does not permit one to produce sintered products with a porosity of less than 1%, then shock compression permits one to lower the porosity to a value of less than 0.5% [7].

Analysis of the results cited above permits the following conclusions to be drawn.

1. In the process of static compression of annealed PN55T45 grade powder at room temperature, martensite transformation takes place to a significant degree with the formation of the

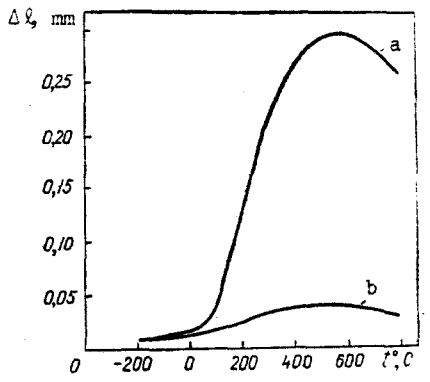

Fig. 3

B19 type phase, in which the volume share of these phases increases with increasing compression pressure up to roughly 1000 MPa.

2. During shock compression, the volume share of the low-temperature phases does not increase substantially. Simultaneously, the lattice defects after shock compression grow by a greater extent than after static compression.

3. When heating pressings made of PN55T45 grade powder, produced by shock compression, the magnitude of their thermal expansion roughly corresponds to the thermal expansion of titanium nickelide at room temprature; the effective thermal expansion of samples produced by static compression is roughly an order of magnitude higher.

4. The noted features of the behavior of titanium mononickelide during mechanical loading and subsequent heating induce, due to the application of shock compression, the capacity to produce denser sintered products made of compositions containing TiNi with a more fine-grained structure and favorable phase composition, since during sintering after shock compression, the pressings are not distended as a result of martensite transformation accompanied by the effect of shape "memory," while the elevated lattice defects activate the sintering process and permit it to be realized at elevated temperatures in the absence of the liquid phase.

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