DOI 10.1007/s11106-017-9871-y
Powder Metallurgy and Metal Ceramics, Vol. 56, Nos. 1-2, May, 2017 (Russian Original Vol. 56, Nos. 1-2, Jan.-Feb., 2017)

MECHANICAL AND FATIGUE PROPERTIES OF POWDER TITANIUM STRIPS, OBTAINED BY ASYMMETRIC ROLLING

K. A. Gogaev,[1] V. S. Voropaev,[1] Yu. N. Podrezov,[1] Yu. F. Lugovskoi,[1,2] V. A. Nazarenko,[1] A. Yu. Koval,[1] and Ya. I. Yevych[1]

UDC 539.385

The mechanical properties of titanium powder strips obtained by asymmetric rolling technique are investigated. It is found out that the use of asymmetric rolling during the consolidating and repeated compacting rolling allows obtaining a strip with better mechanical properties than that obtained by conventional technique. The fracture surface of a titanium strip obtained by symmetric rolling has a significant ratio of the interparticle fracture. After asymmetric rolling, the fracture surface is totally dimpled. It is shown that the asymmetric rolling improves the quality of interparticle contact and, consequently, the ductility and fatigue resistance increase significantly.

Keywords: *powder titanium strips, asymmetric and symmetric rolling, mechanical properties, fatigue resistance.*

INTRODUCTION

Powder techniques for producing titanium and its alloys attract an increasing attention in the international practice and the theoretical aspects of this issue are enthusiastically discussed in the scientific literature. Given the difficulty of obtaining compact products from titanium powder by vacuum hot working, the cold rolling technique of the powder appears to be cost-effective and competitive, compared to other powder techniques. The studies [1–3] consider the techniques for producing compact titanium products by rolling titanium powder and demonstrate the feasibility to create compact titanium strips with acceptable properties.

The results of our studies [4–7] have shown that the asymmetric rolling is more efficient for producing powder products.

Firstly, the hardening parameters increase dramatically in porous samples subjected to asymmetric rolling.

Secondly, along with the improvement of the deformation substructure of the matrix, the final porosity of samples can be reduced significantly.

A further development of this research direction involves obtaining products with a maximum combination of strength and ductility in the strained condition. This research is especially relevant in view of the fact that in recent years the asymmetric rolling is seen as an alternative to the existing techniques of severe plastic deformation (SPD) [8], equal channel angular extrusion (ECAE) [9], and screw extrusion (SE) [10], because this technique is easier to produce sheet products.

[1]Frantsevich Institute for Problems of Materials Science, National Academy of Sciences of Ukraine, Kiev, Ukraine.

[2]To whom correspondence should be addressed; e-mail: lugovskoi_u@ukr.net.

Translated from Poroshkovaya Metallurgiya, Vol. 56, Nos. 1–2 (513), pp. 69–77, 2017. Original article submitted July 16, 2016.

There is no doubt that in terms of the stress–strain distribution, the asymmetric rolling is much more complicated, than the symmetric one. In this case, high mechanical properties can be realized only under strictly regulated deformation conditions. This applies equally to compact titanium products and powder genesis materials.

The purpose of this study is to investigate the effect of the route and process parameters of rolling on mechanical properties and fracture mechanisms under static and cyclic loading of titanium powder strips in a strained state.

MATERIALS AND EXPERIMENTAL PROCEDURE

Titanium powder MTEP (medium titanium electrolytic powder) with a fraction of –02+01 was used. The rolling was performed on a duo-mill using calibrated rolls that allow both symmetric and asymmetric rolling with the same roller gap. Mismatch factor in asymmetric rolling was 1.25

Flat samples (15 mm × 3 mm × 0.35 mm) were cut out to conduct static mechanical tests. The samples were tested for uniaxial tension using a Ceramtest direct stress machine with an automatic loading chart record. The samples cut out along and across the rolling direction were investigated.

The elastic and fatigue properties of small size samples were determined by the resonance method of measurement, while the modulus of elasticity E was determined by exiting flexural modes in the samples. Resonant vibrations were recorded and the modulus of elasticity was determined according to the formula of Kuzmenko proposed in [11]:

$$E = 48\pi\rho l^4 f^2/(H^2\alpha^4), \tag{1}$$

where l is the length of the sample, f is the frequency of proper flexural modes, ρ is the density, H is the thickness of the sample, $\alpha = 1.875$. The modulus of elasticity was determined as an average value based on at least three sample tests. The procedural accuracy error, determined as a sum of the errors of (i) measuring the density of the material, (ii) the frequency of vibrations, and (iii) the geometrics of the sample, was 5%.

The fatigue resistance was studied in bending through an angle of the rod samples with permanent cross-section at a frequency of $f = 2.4$ kHz according to the procedure described in [12]. The maximum stress σ in the critical section of the sample was determined when measuring the amplitude of vibrations at two points: at the end of the sample W_0 and at the fitting location of the sample W_1:

$$\sigma = 2\pi f W_0 \,(3\, E_2\rho_2)^{1/2}\, [U(\alpha x) + PV(\alpha x)], \tag{2}$$

where the coefficient P and the argument of αx functions (Krylov) were determined from equation

$$P = -T(\alpha x) \,/\, U(\alpha x) = [-S(\alpha x) - W_0 \,/\, W_1]/\mathrm{T}(\alpha x), \tag{3}$$

where $\alpha = 3.14$, E_2 and ρ_2 are the modulus of elasticity and the density of the sample material, respectively, x is the distance from the free end of the sample to the point of fracture of the latter; V, U, S, and T are the functions of Krylov.

After the static and dynamic tests, the fracture surface was studied using a Jeol scanning electron microscope fitted with Superprob 723 add-on device.

RESEARCH RESULTS AND DISCUSSION

Our previous studies [3, 13], dedicated to finding the optimal routes for rolling titanium powder into the compact strip, were mainly focused on the contact formation conditions. The processes that allow creating the perfect contact within minimum process operations have been worked out. Optimal sintering conditions and the routes of repeated rolling were analyzed in [3], where it was shown that the perfect contact in the powder strip was formed when sintering at 1000°C.

From two to three additional passes are required in symmetric rolling at all stages of producing 0.4 mm thick strip with mechanical properties not lower, than those of similar products produced by conventional techniques. The study [13] has shown that it is expedient to perform the consolidation of the powder by asymmetric rolling. It has been established that a pre-compression of the rolls allows increasing the green strength of the strips almost by an order of magnitude.

In this study, green powdered titanium strips (1 mm thick) were obtained by processing routes described in [13]. The powder was processed by asymmetric rolling with mismatch factor in the roll diameter 1.25. Additionally, the rolls were pressed against each other with a force of 16 t. The relative density of the strips was 97%. The modulus of elasticity of non-sintered strips was 35–40 GPa, while the green strength was 70–80 MPa. The strips were sintered at 1000°C giving the rise in their relative density from 97 to 99%. After sintering, the mechanical properties of titanium strips are as follows:

Limits, MPa:	
Limit of elasticity σ_{001}	233
Yield point σ_{02}	273.5
Tensile strength σ_t.	349
Elongation $\Delta\delta$, %	30
Constriction at break Ψ, %	55
True strain e	0.6

The results indicate that, after sintering titanium strip, the mechanical and physical contacts are formed for 95% and 60%, respectively.

As fabricated products were subjected to repeated rolling until 0.35 mm thick (ε = 73%) in both symmetric and asymmetric rolling. Table 1 shows the mechanical properties of strained strips.

Noteworthy is that the procedure for determining the modulus of elasticity E of light sheets using resonant flexural vibrations of minimal amplitude is sensitive to the state of their surface layers. Given the procedural error, the differences in the values E of the materials studied are insignificant, except for the strip produced by lengthwise asymmetric rolling. In this case, low modulus of elasticity can be associated with a damage of the external layers of the strip resulting from intensive shear deformations when rolling 73%, which, however, requires further research.

The results confirm the findings of our previous studies [5, 13] about the expediency of the asymmetric rolling for producing powdered titanium strips. Moreover, should the asymmetric rolling be used only during consolidating, the properties close to those of compact strips can be obtained for less compacting rollings, compared to the conventional powder technique [3]. Should the asymmetric rolling be used during both consolidating and compacting rolling stages and subsequent strain rollings, the mechanical properties of the strip exceed not only those of the similar strip produced by the conventional powder technique, but also the properties of compact titanium strips.

The yield point and the modulus of elasticity of the powder strips finally processed by symmetric rolling are slightly inferior to those of the compact sheets that have typical values σ_{02} ~ 700 MPa, E ~ 105 GPa, when comparable strain degree [14]. This is due to the fact that, after sintering, the physical contact in the as fabricated products is not formed and the subsequent low-temperature deformation by symmetric rolling does not result in further improvement. This conclusion is confirmed by the results of the examination of the fracture surface of the samples (Figs. 1*a* and 1*b*). Despite predominant dimple fracture, the fracture surface also demonstrates the traces of pores and the areas of interparticle fracture, indicating the powder genesis of the structure of rolled strips.

During asymmetric rolling, strip properties are slightly higher than those of compact strips, which is associated with a further improvement of the contact during repeated cold rolling caused by shear deformation component, typical for asymmetric rolling. The fracture of the sample tested along the rolling direction (Fig. 1*c*), is virtually totally dimple and intercrystalline. This suggests that the perfect contact is fully formed and the interparticle fracture becomes energetically unfavorable. It is of interest that the sample cut out across the rolling direction tends to the interparticle fracture (Fig. 1*d*). This is probably due to the fact that the shear deformation develops in the rolling direction and a localized shear primarily occurs in the interparticle boundaries, located along the rolling direction.

It should be noted that, in asymmetric rolling, not only the strength, but also ductile properties are higher than those of compact titanium. The studies of Podrezov et al. [14, 15] dedicated to the effect of the deformation degree on the homogenous deformation of titanium have shown that, due to strong structural and crystalline magnetic

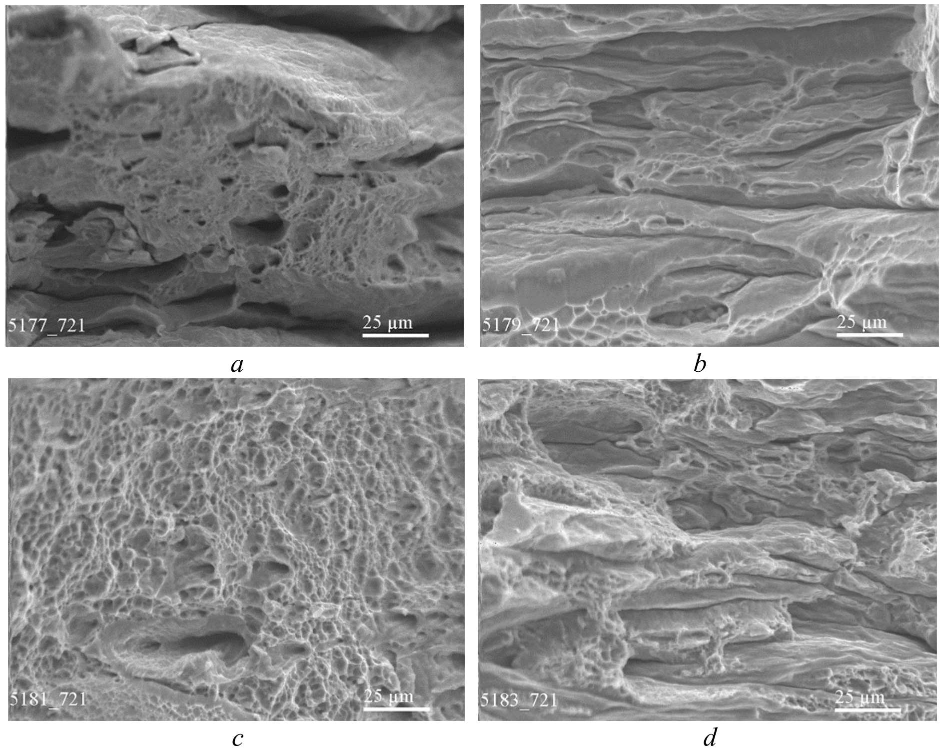

Fig. 1. Fracture surface of powdered titanium strips tested for uniaxial tensile: lengthwise symmetric rolling (*a*), crosswise symmetric rolling (*b*), lengthwise asymmetric rolling (*c*), crosswise asymmetric rolling (*d*)

anisotropy, the homogenous deformation does not exceed 1.5% in heavily deformed titanium sheets. The effect of the homogenous deformation on SPD-produced materials is significantly less and, as Valiev has shown [9], heavily deformed titanium can show a fairly large homogenous deformation in this case.

Zhu et al. [8] have shown that compact titanium, deformed by asymmetric rolling to a high strain degree, demonstrates not only high hardening, but also more homogenous deformation (δ_{homog} = 6%). The data (Table 1) show that, after asymmetric rolling of powdered titanium, the homogenous deformation is higher than after symmetric rolling. For the samples tested lengthwise, its value is close to those obtained in [8], when using compact titanium.

The effect of increased homogenous deformation in the strips obtained by asymmetric rolling is interesting not only from a physical point of view, but also has an applied importance. The study of Vinogradov et al. [16] has shown that it is the SPD that makes SPD-strained materials promising in terms of their fatigue resistance. To check

TABLE 1. Mechanical Properties during Tensile and Cycle Bending of the Strip Rolled of Commercially Pure Titanium Powder

Rolling	σ_t, MPa	σ_{02}, MPa	σ_{001}, MPa	δ_{homog}, %	δ_{total}, %	*E*, GPa	σ_1, MPa
Symmetric:							
Lengthwise	720	670	630	1.45	2.15	98	145
Crosswise	669	618	600	0.23	0.23	95	115
Asymmetric:							
Lengthwise	780	745	719	4.9	7.76	75	155
Crosswise	700	682	655	2.4	2.4	100	130

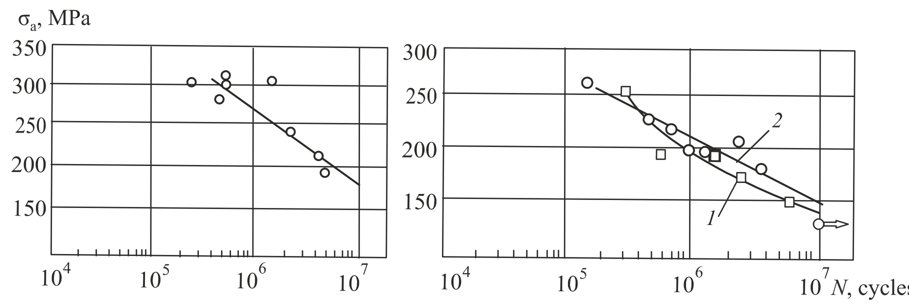

Fig. 2. S–N curves of samples of titanium strips along the rolling direction: cast VT1-0 rolled to 1 mm thickness (*a*), MTEP titanium powder strips obtained by symmetric (*1*) and asymmetric (*2*) rolling with 73% deformation (*b*)

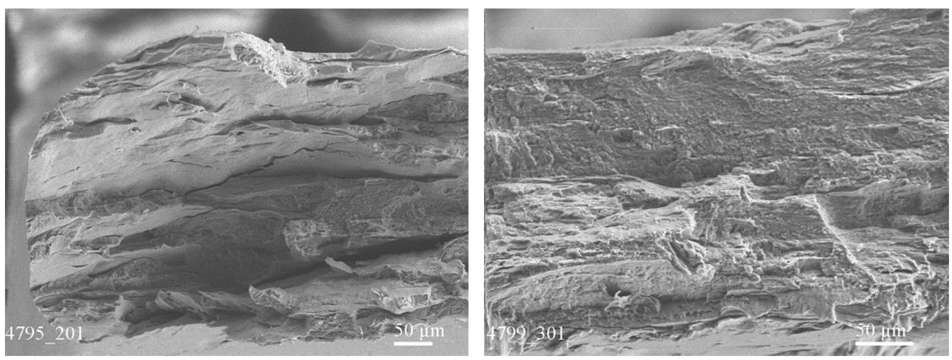

Fig. 3. Fracture surface of powder titanium strips fatigue-tested by lengthwise symmetric (*a*) and asymmetric (*b*) rolling

on this statement, we have performed fatigue tests of powder strips and the results hereof are presented in Table 1. Figures 2*a* and 2*b* show S–N curves for the samples of VT1-0 cast titanium sheet [17] and for the samples of powdered titanium strips, respectively, cut out along the rolling direction.

S–N curves in Fig. 2*b* are located close to each other in all range of the fatigue life and have the same slope with the curve in Fig. 2. However, at high loads, the fatigue resistance of powdered titanium strip increases in symmetric rolling, which is typical for materials that better inhibit the fatigue crack due to structure features. Lengthwise and crosswise rolling data on the fatigue resistance of powdered titanium strips (Table 1) are slightly lower than the similar results for 1 mm thick titanium sheet produced by conventional casting and subsequent cold rolling (Fig. 2*a*). This indicates the possibility of further improvement of titanium strip properties depending on the amount of impurities in the powder, the type and degree of rolling.

Decomposing cracks were identified at the fatigue fractures (Fig. 3) of all samples, mainly in the break areas, whose density increased when moving from asymmetric to symmetric rolling. In the latter case, the fracture propagation mechanism is primarily interparticle, however, these are the samples produced by symmetric rolling that demonstrate low-rate fracture development.

This phenomenon can be explained by the fact that the growing crack is inhibited by material areas with a relatively weak bond between particles in the middle of the sample under the influence of increasing stress concentration in the crack tip.

Therefore, the obtained results indicate the prospects of asymmetric rolling to production powder titanium strips.

CONCLUSIONS

It has been established that the use of asymmetric rolling, when consolidating the strip, allows reducing the number of repeated rolling to one pass as per symmetric route to achieve the mechanical properties comparable to those of the compact tape produced by conventional technique.

It has been established that the use of asymmetric rolling in both consolidating and repeated compacting rolling allows obtaining the strip surpassing the compact strip produced by conventional technique as per the strength and ductility.

It has been found out that the fracture surface of the strip compacted by symmetric rolling shows mostly dimple fracture under tension, however, the traces of pores and interparticle fracture areas indicating the powder genesis of formed structure are observed on it.

It has been found out that the fracture of the strip compacted by asymmetric rolling is virtually dimple and intercrystalline indicating the formation of the perfect contact throughout the product.

Cyclic tests have demonstrated that the modulus of elasticity of the strips compacted by asymmetric and symmetric rolling till 73% deformation is more than 10% below this value for cast titanium alloys.

It has been established that the bending fatigue limit (based on 10^7 cycles) of powder materials measured along and across the rolling direction is also slightly lower than for the VT-1.0 cast alloy.

Fatigue fractures of samples of all materials have revealed that decomposing cracks occur mainly in the break areas under the action of shear stresses in the crack tips, whose density increases when moving from asymmetric to symmetric rolling

REFERENCES

1. M. V. Mal'tsev, V. G. Khromov, and Z. N. Malyshkina, "Study of production and properties of cermet electrolytic titanium," *Trudy Gor. Politekh. Inst.*, **XXVI**, No. 15, 53–58 (1970).
2. L. S. Shmelev and V. K. Sorokin, "Commercial production of sheet titanium by powder rolling," *Tsvet. Metally*, No. 10, 49–55 (1993).
3. K. A. Gogaev, V. A. Nazarenko, V. A. Voropaev, et al., "Mechanical properties of powder titanium at different production stages V. Properties of a titanium strip produced by powder rolling," *Powder Metall. Metal. Ceram.*, **48**, No. 11, 652–658 (2009).
4. K. A. Gogaev, G. Ya. Kalutskii, and V. A. Voropaev, "Asymmetric rolling of metal powders I. Compactability of metal powders in asymmetric rolling," *Powder Metall. Metal. Ceram.*, **48**, No. 3, 152–156 (2009).
5. K. A. Gogaev, V. A. Voropaev, Yu. N. Podrezov, et al., "Production of titanium powder sheets by asymmetric rolling," *Powder Metall. Metal. Ceram.*, **51**, No. 9, 509–517 (2012).
6. V. S. Voropaev, "Effect of mismatch value in asymmetric rolling of powders on the diagram of the total normal contact stress," *Tekhnol. Sistemy*, **4**, No. 61, 28–30 (2012).
7. K. A. Gogaev, G. Ya. Kalutskii, and V. S. Voropaev, "Asymetric rolling of metal powders. II. Angular parameters of asymmetric rolling," *Powder Metall. Metal. Ceram.*, **48**, No. 5, 274–278 (2009).
8. Y. Zhu, X. Wu, M. Yang, et al., "Heterogeneous lamella structure unites ultrafine-grain strength with coarse-grain ductility," *PNAS*, **112**, No. 47, 14501–14505 (2015).
9. R. Z. Valiev and T. G. Langdon, "Principles of equal-channel angular pressing as a processing tool for grain refinement," *Prog. Mater. Sci.*, **51**, 881–981 (2006).
10. Y. Beygelzimmer, V. Varyukhin, and D. Orlov, "On Ultrafine Grained Materials: Properties and Structure," in: *Proc. of the Second Int. Symp. – Los Alamos Nat. Lab. Edition* (2002), pp. 234–238.
11. V. A. Kuz'menko, *Sound and Ultrasound Vibrations during Dynamic Tests of Materials* [in Russian], Izd. AN UkrSSR, Kiev (1963), p. 152.
12. Yu. F. Lugovskoi, "Bending fatigue test procedure for composite materials produced by electron beam evaporation," *Probl. Spets. Elektrometal.*, No. 4, 61–65 (1987).

13. K. A. Gogaev, V. A. Voropaev, Yu. N. Podrezov, et al., "The effect of compacting rolling on the properties of titanium powder mill products," *Powder Metall. Metal. Ceram.*, **55**, No. 11, 633–639 (2017).
14. Yu. N. Podrezov, E. M. Borisovskaya, D. G. Verbilo, and V. A. Pisarenko, "Features of structurization and mechanical properties of deformed titanium," *Fiz. Tekh. Vys. Davl.*, No. 2, 110–118 (2007).
15. Yu. N. Podrezov and V. I. Danilenko, "Analyzing the effect of repeated deformation on the mechanical behavior of screw-extruded titanium samples," *Electron. Mikrosk. Proch. Mater.*, No. 15, 68–79 (2009).
16. A. Vinogradov, T. Ishida, K. Kitagawa, and V. I. Kopylov, "Effect of strain path on structure and mechanical behavior of ultra-fine grain Cu–Cr alloy produced by equal-channel angular pressing," *Acta Mater.*, **53**, No. 8, 2181–2192 (2005).
17. S. A. Firstov, O. P. Ostash, Yu. F. Lugovskii, et al., "Fatigue properties of thermally-deformed Ti–Si alloys," *Fiz. Khim. Mekh. Mater.*, No. 1, 53–59 (2014).