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MECHANICAL PROPERTIES OF POWDER TITANIUM AT DIFFERENT PRODUCTION STAGES. V. PROPERTIES OF A TITANIUM STRIP PRODUCED BY POWDER ROLLING

K. A. Gogaev,[1] V. A. Nazarenko,[1] V. A. Voropaev,[1]
Yu. N. Podrezov,[1,2] D. G. Verbilo,[1] O. S. Koryak,[1] and I. Yu. Okun[1]

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A method for producing strips by titanium powder rolling is proposed. The effect from powder annealing on the porosity of the initial billet is analyzed. The optimal temperatures of sintering and annealing after intermediate rolling are determined. It is shown that, under the optimal deformation conditions, the strip has mechanical properties that compare well with those of strips produced conventionally.

Keywords: *titanium powder, rolling, sintering, annealing, strength, plasticity, quality of contact.*

INTRODUCTION

As pointed out in the previous papers of this series [1–4], the powder technologies of producing titanium and its alloys become more and more popular worldwide and the relevant theoretical aspects are extensively covered in the scientific literature. Since it is difficult to make compact parts from powder titanium by hot working in vacuum [5–6], cold rolling of powders seems to be quite a cheap and competitive method as compared with other processes. The papers [7–12] consider the production of compact titanium parts by titanium powder rolling and demonstrate that a compact titanium strip with adequate properties can be made. However, they either propose too complex conditions (sixfold reduction, fivefold vacuum heating) [11] to reach adequate strength and plasticity or do not justify the pressing and sintering conditions selected but rather focus on the production of long-length parts with adequate properties [12].

This paper considers technological aspects of producing sheet titanium by powder rolling taking into account the data from [3, 4] on the formation of contacts in powder titanium. The objective is to optimize and justify the pressing and annealing conditions for rolled powder titanium products.

PRODUCTION OF SHEET TITANIUM FROM TITANIUM POWDER

The rolling of metal powders substantially differs from that of compact metals, though there are similarities. Metal powders can be rolled vertically and horizontally [7, 8]. Most popular is vertical rolling with the

[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: podrezov@materials.kiev.ua.

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roll axes in the horizontal plane. Gravity feed of powder to the deformation area is most favorable in this case. Little is published about the rolling of powders for making compact strips for different purposes. There are papers focused on producing molybdenum–copper pseudoalloys [13, 14], dispersion-strengthened materials [15, 16], powder compact titanium strips [11, 17, 18], and powder compact iron strips [19, 20].

We established the optimal conditions for rolling titanium powder of fraction –05+01 into a compact strip 0.4 mm thick with mechanical properties comparing well with those produced with conventional methods. The preliminary annealing of the powder decreases its hardness and thus the primary rolling may result in higher density (lower porosity) and reduce the number of rolling passes. Intensive sintering of loose powder begins above 700 ºC; therefore, annealing was conducted at 600 to 700ºC for 1 h in 0.001 Pa vacuum.

The properties of PTÉS titanium powder ensure a wide range of rolling speeds [14]. The empirical ratio between the diameter of the rolls and the thickness of the strip is known: the latter is 1 to 1.5% of the former. We selected horizontal rolling; roll diameter 200 mm and rolling speed 1 m/min. Four strips 1.5 × 100 × 900 mm each were rolled using powders annealed at different temperatures. The edges of the strips were removed, and the remaining part was cut into 90 × 100 mm billets since small samples are more convenient for vacuum sintering. The porosity of each billet was calculated.

The billets were initially sintered at 1000 or 1200°C, which, according to [3], corresponds to temperatures at which physical contact forms in titanium. After sintering, the porosity of the billet was determined again, the billet was first rolled on a 160/100 mm diameter KVARTO rolling mill at room temperature to a thickness of 0.8 mm, and the final thickness and porosity were measured. The samples were further annealed for 1 h at temperatures at which either physical contact (1000 or 1200°C) or mechanical contact (650°C) forms.

At the last stage of the process, the strip was finally rolled to a thickness of 0.4 mm, its porosity was measured, and some part of the billets was subjected to the final thermal treatment—recrystallization annealing (650 or 1000°C, 1 h, vacuum). The porosity was measured with the gravimetric method and hydrostatic weighing.

EXPERIMENTAL PROCEDURE

To optimize the process parameters, we examined the mechanical properties of the samples. For this purpose, the strips produced under different conditions were cut into 0.4 × 10 × 50 mm samples with the long axis aligned with the rolling direction. The resistivity of the samples was measured, and then a spark-cutting machine and special template were used to cut out plane samples for mechanical tests with an effective length of 15 mm, width of 3 mm, and thickness of 0.4 mm. Uniaxial tension tests were performed on an UTM-100 machine, which automatically plotted the stress–strain curve. The fracture surface was examined with a Superprobe 723 scanning microscope.

EXPERIMENTAL RESULTS

Due to the structural genesis of the titanium strip at different production stages, the initial porosity of the billet made of the rolled titanium product is an important parameter influencing the properties of the finished product. The porosity should be minimum. Table 1 summarizes the porosities of 1.5 × 90 × 100 mm plates cut out from different sections of a billet made of powders before and after annealing. As expected, the powder would become softer after annealing and, thus, the porosity of billets made of the annealed powder noticeably lower. This is primarily true for the powder annealed at 700°C. This annealing mode seemed to be the most preferable. However, powder particles partially fuse during this annealing. On the one hand, this complicates the process since grinding is required and, on the other hand, may cause local or macroscopic loosening of the billet.

Sintering at 1200 or 1000°C decreases porosity; this decrease is greater than that in compacts [3], which seems to be associated with nonequiaxial pores in the samples rolled and, as a result, smaller radius. The billets sintered at 1000°C are compacted to somewhat less than those sintered at 1200°C. The final porosity θ (prior to compaction rolling) of annealed powders is 8 to 13%. Green powder is not rolled and sintered so well: $\theta = 14.1\%$.

For subsequent compaction rolling, we selected samples whose porosity after annealing was close to the average porosity of the billet. The only exception was the densest sample made of the powder sintered at 700ºC: its

TABLE 1. Porosity of Powder Billets before and after Sintering at 1200 °C

Annealing temperature, °C	Porosity, %	
	initial	after sintering
Initial	13.7	13.1
	17.0	16.8
	17.0	16.4
	12.0	10.9
	16.0	15.1
	14.85 ± 1.63	14.1 ± 1.41
600	14.0	13.0
	12.2	10.9
	13.4	11.8
	14.7	12.9
	11.8	10.4
	12.9 ± 1.56	11.7 ± 1.84
650	11.4	10.1
	12.0	11.1
	9.8	8.5
	10.1	9.1
	11.6	11.0
	11.5 ± 0.14	10.55 ± 0.64
700	7.8	3.8
	10.2	8.5
	11.1	9.0
	8.7	6.1
	10.2	9.1
	9 ± 1.69	6.45 ± 3.75
700	21.0	18.7

TABLE 2. Annealing Temperature and Porosity of Samples at Different Stages of Titanium Strip Production

Sample No.	Annealing temperature, °C	Initial porosity of the billet, %	Sintering temperature, °C	Porosity after sintering, %	Porosity after the first rolling, %	Temperature of intermediate annealing, °C	Porosity after the second intermediate rolling, %	Annealing temperature of the strip, °C
618	600	14.7	1200	12.9	<3	1200	<1	650
629-1	600	17.0	1200	15.8	<3	1200	<1	650
629-2	600	17.0	1200	15.8	<3	1200	<1	1000
652	650	12.0	1200	11.1		1200	<1	650
61′	600	16.0	1000	8.0	3.9	650	<1	650
63′	600	8.9	1000	7.2	5.1	650	<1	650
63 (h = 0.8)	600	8.9	1000	7.2	5.1	650	<1	650
79	700	7.8	1200	3.8		650	<1	650
8	No	13.7	1000	13.1	9.5	650	<1	650
61	600	13.0	1000	8.0	3.9	650	<1	–
63 (h = 0.4)	600	8.9	1000	7.2	5.1	650	<1	–
611	600	14.4	1200	11.7	–	–	<1	
629	600	17.0	1200	15.8	<3	1200	<1	–
650	600	8.5	1000	8.3	–	–	<1	–

porosity after sintering at 1200°C was just 3.5%. The strip was rolled in different modes. Table 2 summarizes the thicknesses of the strip after rolling and intermediate sintering modes for each sample; there are also temperatures of the final annealing (if any). This variety of processes for making strips allowed us to compare the rolled products in mechanical characteristics with each other and with similar materials produced with conventional methods.

To compare the quality of the strips, samples for mechanical tests were cut out from them (the direction of the long axis of the sample aligned with the rolling direction). Two samples of each strip were tested; additional samples were tested when the results greatly differed. Tables 3 and 4 summarize the yield strength (σ_y), ultimate strength ($\sigma_{u.s}$), true breaking stress ($\sigma_{b.s}$), uniform strain (ε_u), true strain-to-failure (e_r), and reduction (ψ) of the strips tested in recrystallized and deformed states. For reference, Tables 5 and 6 provide results from mechanical tests of VT1-0 recrystallized titanium and data from [4] for VT1-0 titanium rolled to different strains (e_r is the true strain of rolled samples). Samples 3 and 4 (Table 6) are most appropriate for strain comparison since the powder billets were finally compacted with double reduction (from 0.8 to 0.4 mm).

All mechanical properties in the Tables are structure-sensitive. Hence, comparing the results permits not only assessing the quality of the material but also identifying the nature of structural imperfection. Note, however, that the shape of samples reported in the cited papers (cylinders 3 mm in diameter) differs from that described above in the experimental procedure, the testing conditions being the same (tension at room temperature at a rate of 10^{-3} sec^{-1}). This difference may reveal itself in the comparison of ultimate mechanical characteristics since thin hardened samples fail by shear at 45º.

DISCUSSION OF RESULTS

Table 2 shows that sample 650 was produced in the simplest rolling conditions. The strip was rolled from a billet up to 0.4 mm in thickness without intermediate sintering. The mechanical properties show that these conditions do not permit producing a high-quality strip. The sample rolled in these conditions has lower yield strength and uniform strain than the compact material does and very low strain-to-failure. There are delamination areas and failure fragments of unsintered particles on the fracture surface (Fig. 1a).

TABLE 3. Mechanical Properties of Annealed Titanium Strips Produced in Different Modes

Billet No.	σ_y, MPa	$\sigma_{u.s}$, MPa	ε_u, %	$\sigma_{b.s}$, MPa	e_r	ψ, %
618	333.1	469.2	16.9	761.7	0.850	57.3
629-1	314.3	451.2	15.0	845.8	1.012	64.0
629-2	297.3	402.1	8.1	265.3	0.179	16.4
652	340.5	478.0	12.7	812.3	0.945	60.3
61′	340.9	485.5	13.5	725.4	0.747	52.6
63′	350.1	490.4	12.5	799.9	0.641	48.5
63 (h = 0.8)	333.0	444.8	10.6	465.8	0.207	18.7
79	304.7	442.1	17.9	910.0	1.139	68.9

TABLE 4. Mechanical Properties of Rolled Titanium Strips Produced in Different Modes

Billet No.	σ_y, MPa	$\sigma_{u.s}$, MPa	ε_u, %	$\sigma_{b.s}$, MPa	e_r	ψ, %
8	754.3	815.7	1.5	1078.7	0.469	39.4
61	592.8	725.8	1.6	871.3	0.391	33.7
63 (h = 0.4)	708.8	762.7	1.9	954.8	0.337	29.5
611	490.4	818.8	2.0	1015.2	0.346	29.3
629	637.0	711.1	1.3	1001.9	0.536	41.0
650	295.6	409.1	1.4	392.8	0.211	20.6

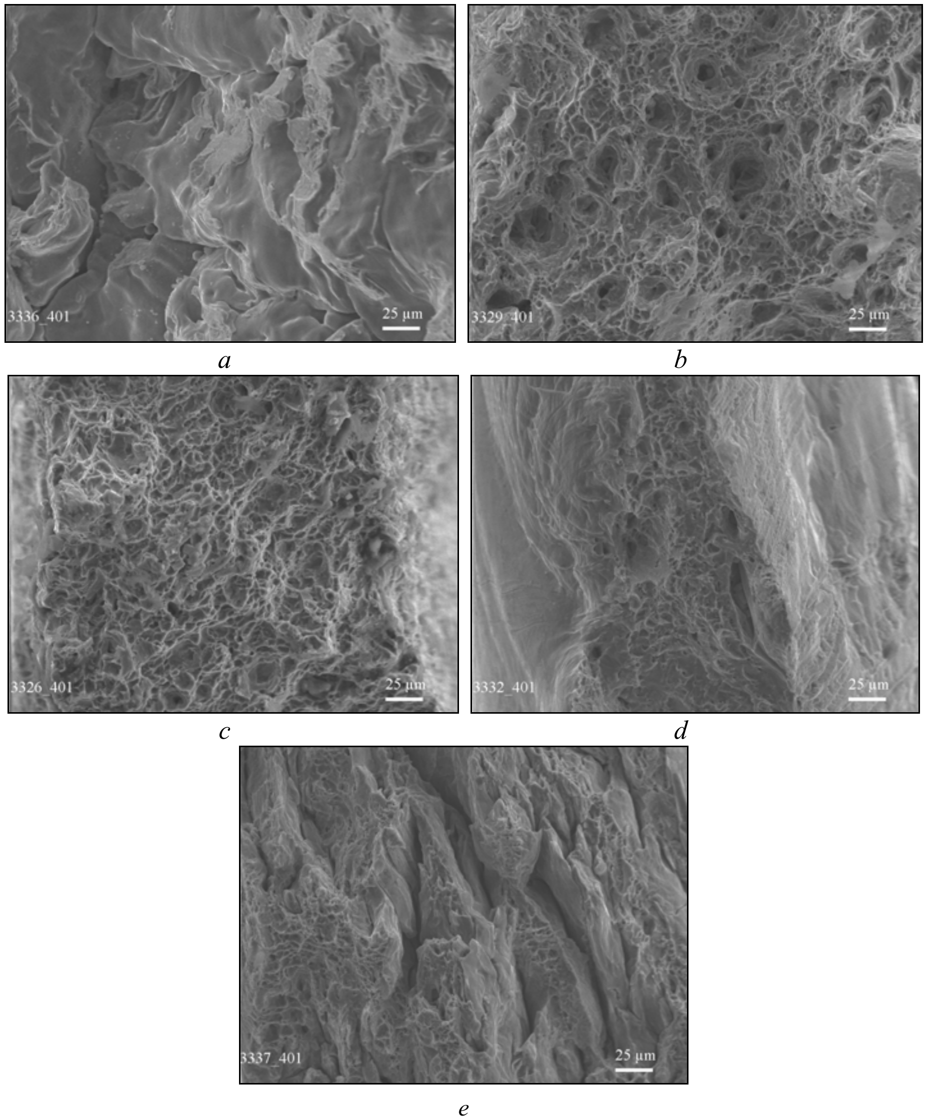

Fig. 1. Fracture patterns of samples 650 (*a*), 79 (*b*), 652 (*c*), 629-2 (*d*), and 63 (*e*) cut out from strips

The strip from the densest billet (sample 79) was made with two intermediate rolling passes; low-temperature annealing (650°C) was conducted after each pass. The data presented in Table 3 show that the strip has proper quality. Its yield strength, uniform strain, and strain-to-failure are comparable with the properties of compact recrystallized titanium. Intracrystalline dimple fracture is observed (Fig. 1b).

The samples made from more porous billets 618, 629, and 652 were also obtained in two rolling passes but had higher temperature of intermediate annealing (1200ºC). After the final annealing at 650ºC, the properties of these billets are also comparable with those of a compact strip; dimple fracture is observed, without apparent traces of residual interparticle delamination (Fig. 1c).

Sample 629-2 was rolled in the same conditions but subjected to final high-temperature annealing (1000ºC). This annealing greatly intensified grain growth and delamination at grain boundaries (Fig. 1d), abruptly decreased plastic strain-to-failure, and somewhat decreased yield strength and uniform strain.

The strips from samples 8, 61, and 63 were rolled in three passes, but sintering and intermediate annealing were conducted at low temperatures: primary sintering at 1000ºC and intermediate annealing at 650ºC. The results of mechanical tests showed that these conditions were less efficient for making a strip. Although these samples have comparable yield strength, their uniform strain and strain-to-failure are much lower; this is caused by process-induced defects that affect mechanical properties.

TABLE 5. Mechanical Properties of VT1-0 Recrystallized Titanium Annealed at Different Temperatures

T, °C	σ_y, MPa	$\sigma_{u.s}$, MPa	ε_u, %	$\sigma_{b.s.}$, MPa	e_r	ψ, %
Initial	350	463	20	830	0.82	61
600	290	400	17.2	780	0.91	64
650	340	420	17.6	815	0.85	62
700	310	405	17.5	750	0.91	63

TABLE 6. Mechanical Properties of Titanium VT1-0 Rolled to Different Strains

Sample No.	e_{roll}	σ_y, MPa	$\sigma_{u.s}$, MPa	ε_u, %	$\sigma_{b.s.}$, MPa	e_r	ψ, %
1	1.60	655	700	1.6	1442	1.378	74.79
2	1.25	675	712	1.7	1521	1.489	77.46
3	1.00	660	685	1.0	1489	1.482	77.30
4	0.70	585	590	0.9	1403	1.409	75.56
5	0.50	575	619	1.0	1254	1.371	74.60
6	0.35	527	548	1.5	1342	1.495	77.60
7	0.20	493	523	1.8	1298	1.801	83.50

Table 2 shows that most strips were annealed at low temperature (650ºC) after the last rolling pass. Samples 61, 63, and 629 were both annealed and pressed, while sample 8 only pressed. Comparison of the experimental results shows that the yield strength of pressed strips almost doubles (from 350 to 700 MPa) as compared with annealed ones, but uniform strain decreases by almost an order of magnitude (from 15 to 1.5%). Note that this behavior is also typical of conventionally produced sheets. Table 6 shows that these indicators for strips produced with powder metallurgy methods are not worse than those for conventionally produced titanium sheets.

Only does the true strain-to-failure e_r substantially differ. As noted previously, the samples have different shapes and our data cannot be compared with those in Table 6. Hence, we will carry out a comparative analysis of this indicator for strips produced with different methods. Tables 3 and 4 show that sample 629 has the highest true strain in the initial and recrystallized state. The strain-to-failure of samples 61 and 63 is about 33% lower. There are typical delamination areas over pressed sample 63 (Fig. 1e), which are caused by the inadequate quality of the strip produced by low-temperature sintering and intermediate annealing.

CONCLUSIONS

A relationship between the fracture pattern and mechanical properties of a powder titanium strip is established. Samples with the highest true strain-to-failure e_r—a parameter that characterizes the quality of physical contact—show intercrystalline dimple fracture. Dimple fracture is observed in samples sintered at the temperature of formation of physical contact. A powder billet should be sintered at the same temperature.

Preliminary annealing of titanium powder decreases the content of oxygen and eliminates cold hardening of the powder and thus permits obtaining dense billets in the primary rolling.

The final annealing of the strip should be conducted at temperature no higher than 650°C. At higher annealing temperatures, the grains start growing and deteriorate the properties of the strip.

To produce a high-quality titanium strip, two intermediate annealings should be conducted at the temperature of formation of physical contact in titanium. In this case, the properties of the product are as good as those of conventionally produced strips.

To decrease the number of intermediate rolling passes and intermediate annealings, the porosity of powder billets should be lower than 10%.

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