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HOMOGENEITY OF TITANIUM MANUFACTURED BY SOLID STATE CHIPS RECYCLING

Krzysztof Topolski
Faculty of Materials Science and Engineering, Warsaw University of Technology, Warsaw, Poland
kt.topolski@gmail.com

Abstract

This work presents a promising method for recycling chips, carried out in the solid-state with the use of plastic working. High-purity (hp) titanium 99.99 chips after milling were subjected to a three-step procedure to transform these chips into a solid material. The objective of this work is to demonstrate the potential of this new processing method and to present the results of experiments conducted to determine the homogeneity of the final product. It was found that the processing method employed resulted in the formation of a solid and consolidated product in the form of a rod 8mm in diameter and 700mm long. The rod exhibited a grained microstructure typical of the Ti in the as-received state. Furthermore, the mechanical properties of the rod-product were also similar to those of titanium in the as-received state. For example, the hardness of the manufactured rod (average 97 HV10) was typical of the hardness of commercial Ti hp (94 HV10). In addition, the product obtained was characterized by adequate homogeneity in terms of its microstructure and mechanical properties. It was also found that the recycled material is as homogeneous as the commercial, reference Ti in the as-received state.

Keywords

Homogeneity, Titanium, Chips, Recycling, Structure, Properties

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1. Introduction

The basic and most common method of recycling metallic chips is re-melting. However, due to the high energy consumption and necessity to protect the materials from contact with the air, alternative methods have also been sought and developed. In this area, solid-state processing of metallic chips using plastic working (without re-melting) is one such alternative. This technology of scrap recycling seems to be promising and reasonable.

It has been proved that this method is effective and makes it possible to transform chips into solid and consolidated materials. The literature on this subject contains a lot of satisfying results, what is the evidence of positive effects of these experiments. Most such investigations concern aluminium and its alloys (Shamsudin et al., 2016, Bingbing et al., 2017) or magnesium and its alloys (Peng et al., 2008). Fewer studies have dealt with the processing of titanium (Luo et al., 2010). In the context of the specific method applied, conventional direct hot extrusion dominates (Wen et al., 2010). Other methods used included equal channel angular pressing (ECAP, Lapovok et al., 2014) and high-pressure torsion (HPT, Zhilyaev et al., 2008).

For recycled material to meet the quality requirements that qualify it as usable, it must exhibit an appropriate macrostructure, microstructure and mechanical properties in entire of its volume. Therefore, it means that the structural homogeneity and the homogeneous properties of the manufactured product are of critical importance. The recycled material should be homogeneous in microstructure and its properties. They should be the same or similar to those of the analogous commercial material available on the market. Comparing a recycled product with the same original material in the as-received state is the simplest and most reasonable method of verifying whether these quality requirements have been met.

2. Motivation, Aim and Scope of the Research

In this work, unconventional plastic working was applied as a new method of recycling titanium chips. The processing was conducted without re-melting phenomena. As usual in the case of novel experiments and materials, certain fundamental questions have to be looked into, concerning, for example, the homogeneity of the resulting product. This problem is important, for example, in the context of the potential application of such new material. Although in many studies relatively volumetric products have been obtained, the problem of homogeneity has not been studied in detail in the literature data before. Instead of this, the literature data reported about the issues such as chips microstructure, tool wear, machinability (Nouari et al., 2014), shearing of machined material during machining (Kent et al., 2018), or grain orientation and grain boundaries in the recycled microstructures (Luo, 2020).

Therefore, this study aimed to investigate the potential of this new processing method as well as to examine and analyze the homogeneity of the product obtained.

The issue of homogeneity was analyzed based on research on the microstructure and mechanical properties of the final product. The scope of the research included analysis of grain size, hardness, microhardness, indentation tracks, yield stress, tensile strength and elongation.

The final product of recycling was in the form of a rod 8mm in diameter and 700mm long. Such a material can be considered to be a relatively bulk and volumetric material, making the research on homogeneity fully reasonable.

3. Materials Processed and Investigated

The material examined was single-phase high purity titanium (Ti hp) 99.99 wt. %. This grade, among all grades of pure titanium, exhibits the highest possible purity and plasticity and is the softest. The chemical composition of this material, evaluated based on the certificate data, is given in Table 1.

In the as-received state, the titanium was in the form of a rod with a diameter of Ø50mm. This rod was subjected to machining (milling) to obtain the specific and required type of chips: short, flat, loose, coarse chips with a thickness of about 0.3-0.4mm. It should be explained that the titanium Ø50mm in the as-received state was also used as a reference material to be compared with the material obtained by recycling (rod Ø8mm).

Table 1: *Chemical Composition of Investigated High Purity Titanium 99.99wt.%*

Element	**O**	**N**	**C**	**H**	**Fe**	**Si**	**Ca**
[%]weight	<0,032	0,00088	<0,0005	<0,0001	0,00026	0,000038	<0,00005
Element	**Cr**	**Cu**	**Al**	**Mn**	**Ni**	**Cl**	**Ti**
[%]weight	0,000048	0,000043	0,000029	0,000023	0,000014	0,00002	Base

(Source: Original Author's Results)

4. Recycling Procedure

To recycle the chips without re-melting into solid rod-product, a special process in the solid-state was applied, which consisted of: (1) preliminary consolidation of the chips into a “briquette”, carried out by the upsetting method, (2) heating of the briquette to a temperature of 350°C and annealing at that temperature for 20 minutes, (3) final extrusion of the briquette using an unconventional plastic working method. This method was the direct extrusion with a rotating die (rotated cyclically, and reversibly in both directions). The process resulted in a rod 8mm in diameter and about 700mm long. More information about this method and processing procedure analogous to those applied in this work can be found in the reference (Topolski et al., 2021).

5. Methods of Investigation

In this work, homogeneity was analyzed by evaluating the microstructure of the material, and by the hardness measurements, microhardness measurements, compression tests and tensile tests. The method of obtaining the samples is presented in Fig.1, which shows, at the same time, a macroscopic picture of an actual rod product. The mechanical tests were conducted using two samples taken from two different areas of the rod, which made it possible to examine the homogeneity of the length of the product.

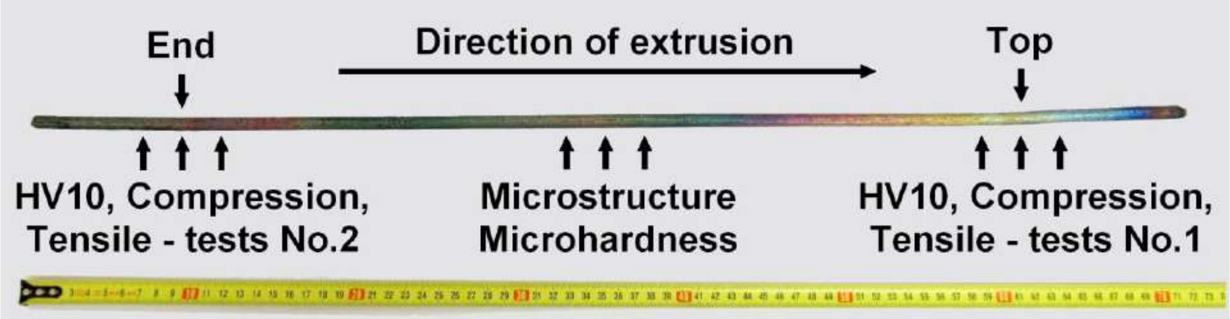

Figure 1: *Macroscopic view of the Recycled Rod-Product with Methodology of Testing (Source: Self Designed, Original Author's Results)*

The microstructure was analyzed using light microscopy, based on the following stereological parameters: (1) average grain size E(d_2) (i.e. the average value (E) of the average equivalent grain diameters (d_2)), (2) SD – standard deviation, (3) CV – coefficient of variation, “α” shape factor, and (5) “β” shape factors. The "α" shape factors determine the deviation of the shape of grain from that of a circle, while the "β" shape factors determine the degree of development of the grain boundary surface. In the case of recycled Ti, in the transverse section, the entire area of the sample was analyzed (circle Ø8mm). In the longitudinal section, the entire area of the sample section was analyzed, where the tested surface was rectangular, with sides 8mm x 10mm. In each case, approximately one hundred grains were used for the stereological analysis.

The hardness measurements were performed using the Vickers method under a load of 10 kg (HV10). Two tests were performed with the use of two different samples taken from different areas (Fig.1). The microhardness measurements were performed using the Vickers method, under a load of 200g (HV0.2). In the transverse sections (circular shape of section), the measurements were carried out along two perpendicular axes (diameters), which made it possible to conduct a more detailed examination of the homogeneity of the material (isotropy). In the longitudinal section (square shape of section), the measurements were carried out along one line constituting the main axis. Using particular measurement points, a hardness distribution diagram was created and the average arithmetic value of hardness was calculated.

The uniaxial tensile tests were conducted at room temperature with a strain rate of $\acute{\varepsilon} = 3.3 \mathrm{x} 10^{-4}$ [1/s], using a vertical MTS 858 universal hydraulic testing machine. The samples had a standard cylindrical shape (circular transverse sections) and were 3mm in gauge diameter and 15mm in gauge length. Two tests were conducted using two samples taken from different edges of the rod (Fig.1). The compression tests were conducted at room temperature with a strain rate of $\acute{\varepsilon} = 1.0 \mathrm{~x~} 10^{-3}$ [1/s]. The same testing machine as in the case of tensile was used. The samples had the standard cylindrical shape and were 7mm in diameter and 10.5mm high. Two tests were conducted using two samples taken from different edges of the rod (Fig.1).

6. Results and Discussion

The examination of the homogeneity was realized based on the following research: (1) microstructure, (2) hardness, (3) microhardness, (4) compression tests and (5) tensile tests.

6.1. Homogeneity of the Microstructure

The microstructure of the reference titanium hp (in the as-received state) was the same in both the transverse (Fig.2a) and longitudinal sections (Fig.2b). Equiaxial, grains with regular shapes were observed in both sections. At the same time, these grains were different in size (from 40-120µm). In the transverse section, the average grain size was 65µm, while in the longitudinal section it was 63µm (see Table 2).

The recycled titanium exhibited a very homogeneous microstructure with equiaxed, similar grains (Fig.3). Over the entire tested surface of each section, there were grains of very similar size and shape, which was confirmed by the small values of the SD and CV coefficients and the similar values of the α and β parameters, which were close to "1". There were no microstructure differences in different regions of the sample. Moreover, it was found that the microstructure was the same in both sections. It is worth noticing that, on the longitudinal sections (Fig.3b), no effect of texture or elongated grains was observed, which often occurs in the products such as the rods.

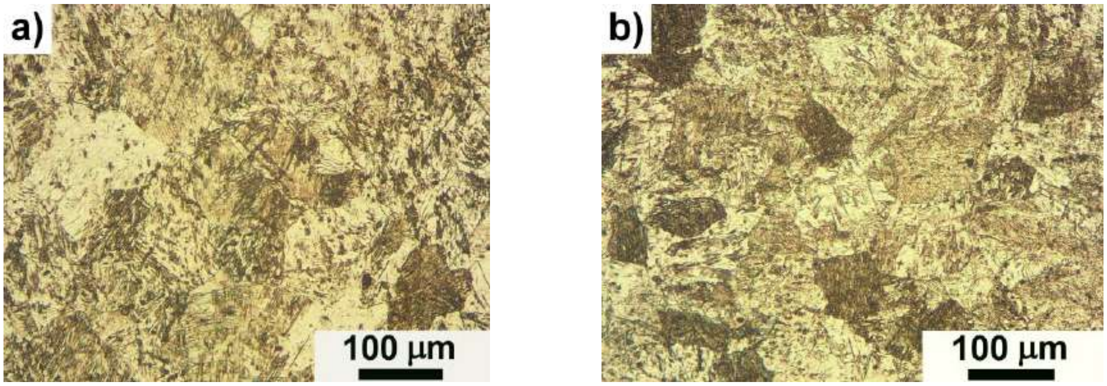

Figure 2: *Microstructures of Ti hp in the As-Received State, Light Microscopy:*
(a) Transverse Section, (b) Longitudinal Section
(Source: Original Author's Results)

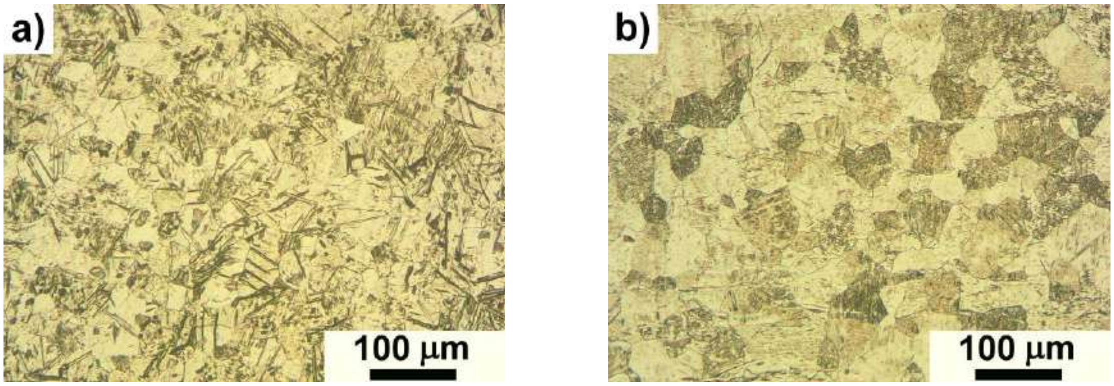

Figure 3: *Microstructures of Recycled Ti hp after Chips Processing, Light Microscopy:*
(a) Transverse section, (b) Longitudinal section
(Source: Original Author's Results)

From the results obtained, it is evident that the resulting microstructure is very homogeneous. In comparison with the microstructure of the commercial Ti in the as-received state, the microstructure of the recycled product is similar but exhibits grains that are about twice smaller and more regular (Table 2). Regardless of these factors, the homogeneity of the recycled Ti is similar to the homogeneity of commercial Ti, which is a very satisfactory result.

Table 2: *Results of the Stereological Analysis of the Examined Microstructures: Ti hp in the As-Received State and Ti hp after Chips Processing*

Sample	Transverse Section					Longitudinal Section				
	$E(d_2)$ [μm]	$SD(d_2)$	$CV(d_2)$	α	β	$E(d_2)$ [μm]	$SD(d_2)$	$CV(d_2)$	α	β
Ti hp as the rec.	65.40	18.96	0.29	1.40	1.27	62.91	18.43	0.29	1.37	1.21
Processed Ti	33.28	10.40	0.31	1.24	1.15	29.86	10.03	0.34	1.23	1.14

(Source: Original Author's Results)

6.2. Hardness Measurements

Despite the fact, that the samples were cut off from different areas of the rod (opposite edges), the hardness values obtained are very similar (Table 3). Hence, the results of the HV10 measurements indicate that the rod-product is homogeneous along its length. In addition, the hardness of recycled Ti is similar (slightly higher due to the smaller grains) to this obtained for the commercial Ti in the as-received state. This confirms that the hardness of recycled material is typical and correct.

During the HV10 measurements, indentation tracks were formed. Microscopic pictures of these tracks are presented in Fig.4. From our observations, it was found that both tracks were symmetrical (both diagonals about 0.5mm). Therefore, it was shown that the rod-product exhibited homogeneity of mechanical properties (isotropy).

Table 3: *Results of Measurements of HV10 Hardness Obtained for Ti hp in the As-Received State and Recycled Ti after Chips Processing, where "SD" is Standard Deviation*

Sample	Ti hp as received			Ti hp after processing		
	test No.1	test No.2	SD	test No.1	test No.2	SD
Hardness HV10	94	93	0.5	99	95	2.0

(Source: Original Author's Results)

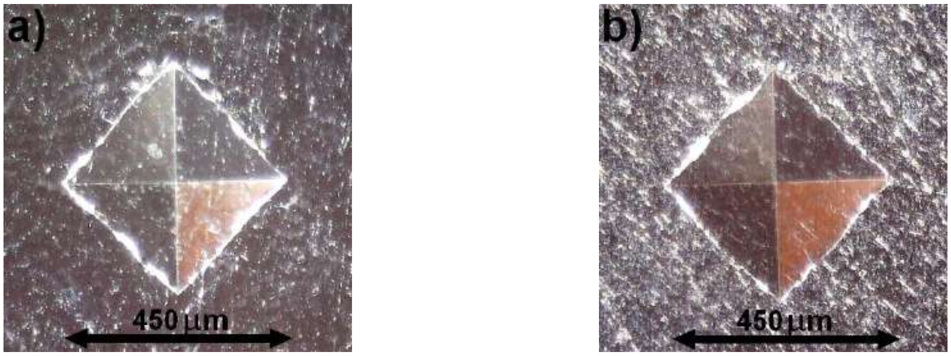

Figure 4: *Microscopic Pictures of HV10 Indentation Tracks Obtained for the Rod after the Chips Recycling: (a) test No.1 (Top of the Rod); (b) test No.2 (End of the Rod)*
(Source: Self Designed, Original Author's Results)

6.3. Microhardness Measurements

The results calculated for all of the investigated samples are presented in Table 4, while, diagrams of the hardness distribution for the recycled Ti are given in Fig.5.

In the transverse section of the recycled titanium (when comparing the microhardness distribution and its average value along two perpendicular axes-diameters) it was found that the results were similar and the hardness did not change significantly. This indicates a high homogeneity (isotropy) of the mechanical properties of the recycled material. Comparing both sections with each other, it was observed that they were similar. Thus, this material can be considered homogeneous within the entire tested volume.

The results also show that the microhardness of the recycled material is similar (minimally and insignificantly lower) to the microhardness of titanium in the as-received state available commercially. In practice, they can be considered the same. Therefore, the hardness values obtained for recycled Ti should be considered to be in the typical and correct range.

Table 4: *Results of the HV0.2 Microhardness Measurements of Ti hp in the As-Received State and Recycled Ti hp After Chips Processing (SD – Standard Deviation, CV – Coefficient of Variation)*

Section, measurement	**Ti hp as received**			**Ti hp after processing**		
	Average	**SD**	**CV**	**Average**	**SD**	**CV**
Transverse, diameter 1	118	7.77	0.07	112	6.86	0.06
Transverse, diameter 2	121	6.34	0.05	114	5.06	0.04
Longitudinal	121	6.46	0.05	118	6.15	0.05

(Source: Original author's results)

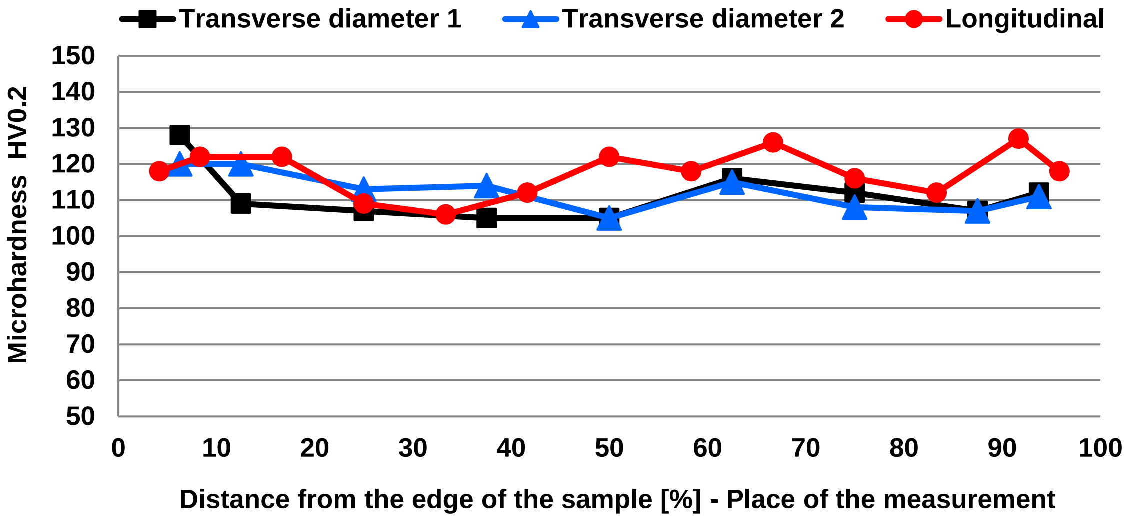

Figure 5: *Microhardness Distribution Obtained for Recycled Ti hp: Transverse Section (Along two Perpendicular Diameters) and Longitudinal Section. The relation between Microhardness-Distance from Edge of Sample (where: 0% - one edge, 50% - Place in the Center, 100% - Opposite Edge)*
(Source: Self Designed by Microsoft Excel, Original Author's Results)

6.4. Compression Tests

The results of this investigation showed that two different samples taken from different areas of the rod product exhibited the same plasticity and strength. The two compression curves are coincident (Fig.6). Moreover, the values of yield stress calculated are the same (Table 5). Therefore, along its length, the material investigated can be considered homogeneous. The graphs show a gradual strengthening of the materials and a proper course of deformation phenomena. During the tests, the relatively large deformation value was achieved ($\varepsilon = 50\%$, reduction of the initial height of the sample by half). Despite this, none of the samples was destroyed (defragmented) into separate parts. Hence, the compressive strength of the material was not calculated. At the same time, it is information that the material after chip consolidation was durable and coherent.

For comparison, the material in the as-received state was also tested. The results show that the mechanical properties and homogeneity of the recycled Ti are correct since they achieved a level typical for commercially available Ti hp in the as-received state.

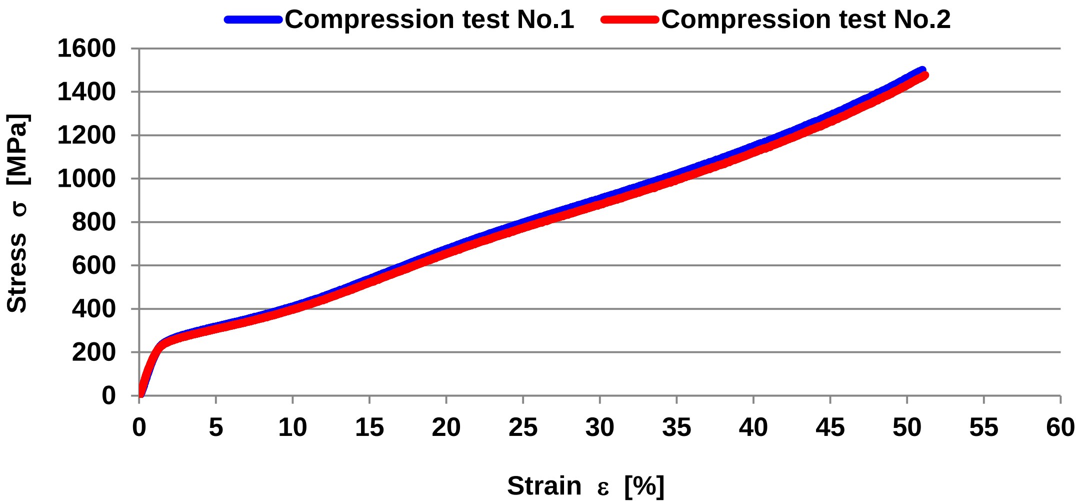

Figure 6: *Strain-Stress Curves Obtained from the Compression Tests. Ti Rod-Product after Processing. Test No.1 Refers to the "Top", and Test No.2 Refers to the "End" of the Rod (Source: Self Designed By Microsoft Excel, Original Author's Results)*

Table 5: *Mechanical Properties Evaluated from the Uniaxial Compression Tests*

Sample	**Ti hp as received**		**Ti hp after processing**	
Test No.	**Test No.1**	**Test No.2**	**Test No.1**	**Test No.2**
Yield stress $\sigma_{0.2}$ [MPa]	241	232	224	216

(Source: Original Author's Results)

6.5. Tensile Tests

The results of the tensile tests indicated that two different samples taken from significantly different areas exhibited the same strength and similar plasticity. The calculated values of the mechanical parameters are similar, which is a very satisfactory result (Table 6). The two strain-stress diagrams (representing two different areas) are coincident for the most part. Although they differ in elongation, both samples exhibit the same, very high elongation (Fig.7). Thus, it can be concluded that the material investigated was homogeneous along its length. Moreover, the repeatability of the values of mechanical properties was similar for both the recycled and as-received titanium. Furthermore, the spread of the results obtained for recycled Ti is less than the spread for Ti in the as-received state. Thus, this confirms that, in the context of tensile tests, material obtained by chips consolidation is homogeneous. It is possible, that the results of individual tests may differ slightly because the properties of materials are within a certain range.

It should be clarified that, during tensile tests, structural defects such as porosity exhibit a strong impact on the mechanical properties and potential applications of manufactured materials. However, in this work, based on the tomography computer research (realized at a resolution of 25 µm - detection threshold) it was found, the porosity of the rod-product was 0.00%, thus the same as for material in the as-received state.

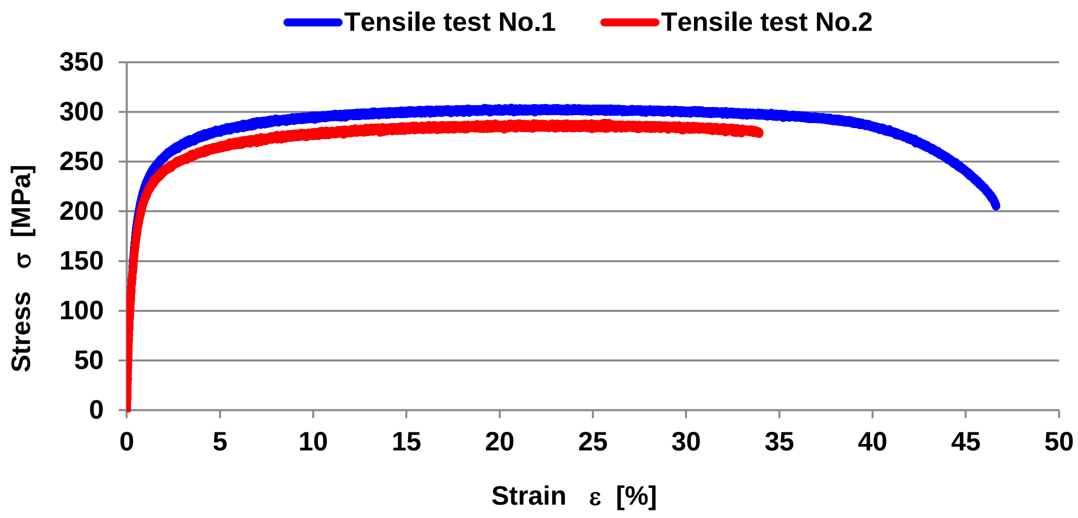

Figure 7: *Strain-Stress Curves Obtained from the Tensile Tests. Ti Rod-Product after Processing, where Test No.1 Refers to the "Top", and Test No.2 Refers to the "End" of the Rod (Source: Self Designed By Microsoft Excel, Original Author's Results)*

Table 6: *Mechanical Properties Evaluated from the Uniaxial Tensile Tests*

Sample	**Ti as received**		**Ti after processing**	
Test No.	**Test No.1**	**Test No.2**	**Test No.1**	**Test No.2**
Yield stress $\sigma_{0.2}$ [MPa]	187	204	192	177
Tensile strength σ_m [MPa]	240	269	304	289
Elongation to failure E [%]	31.4	45.1	46.3	33.7

(Source: Original Author's Results)

7. Summary and Conclusion

In this work, a rod-product 8mm in diameter and about 700mm long was manufactured. With such a relatively bulk and volumetric material available, research was conducted on its structural homogeneity and homogeneity of mechanical properties. These results allow for a positive evaluation of the obtained product and the technology applied. It was found that:

1. The material after recycling was characterized by the same, homogeneous microstructure along its entire transverse section and its entire longitudinal section intended for observation.
2. The microstructures along both the transverse and longitudinal sections were the same, meaning that the material is three-dimensionally homogeneous.
3. Despite the distance separating the "top" and "end" areas of investigation, the properties of samples were the same or similar. Hence, along its length, the rod-product exhibits homogeneous mechanical properties, as was confirmed by the hardness, compression and tensile tests.
4. Based on the indentation tracks and microhardness distribution it was found that the titanium manufactured in this processing is isotropic.
5. The results indicate that the rod manufactured was good quality, semi-finished product. Its microstructure and mechanical properties were homogeneous and analogous to those of commercial titanium available in the trade.

The presented topic of processing high purity titanium chips has been completed successfully and can be considered as perspective and promising. In the context of future research, it is planned to extend the experiments to other chip geometries as well as other titanium grades. An interesting challenge will also be the attempt to process tough titanium alloys, e.g. the frequently used Ti6Al4V.

However, in the scope of the recycling of solid-state chips, there are some limitations and risks. These refer to the energy capabilities of the device and tribological wear of dies.

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