Numerical analysis of metallographic preparation effect on the hardness of titanium alloy

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Abstract: *Titanium alloys due to their good properties are increasingly used in biomedicine. However, in order to improve certain properties, titanium-based alloys with new chemical compositions are designed. In order to be characterized in a satisfactory manner, they must first be adequately prepared. In this paper the two most influential parameters were varied: grinding time and force, while the speed of rotation of the grinding wheel was constant. After grinding with the highest gradation of grind paper, the samples were observed under a light microscope to determine the condition of the surface. Then their hardness was determined by the Vickers method with different indenter loads. After that, the samples were polished under the same conditions, and their hardness was determined again. The obtained hardness values were numerically analyzed and the corresponding functional dependences of the measured hardness on the grinding parameters (time and force) and on the indentation force were determined.*

Keywords*: TITANIUM ALLOY, METALLOGRAPHIC PREPARATION, HARDNESS, VICKERS METHOD*

1. Introduction

Musculoskeletal disorders are the most common health problems. It is estimated that about 90% of the world's population over the age of 40 suffers from degenerative diseases such as arthritis, and their number in the total population has been increasing significantly recently. Biomaterials are solutions to such problems, because their surgical implantation helps to restore the function of otherwise functionally endangered structures [1].

Biomaterials are artificial or natural materials which are used to make implants for the purpose of replacing a damaged or diseased structure with the aim of restoring its original shape and function. Biomaterials are used in various parts of the human body and are implanted as stents in blood vessels, dental implants, orthopedic implants in joints, knees, hips, elbows, ears.

Ever since it was first recognized in 1969 at Clemson University in South Carolina, the field of biomaterials has continued to receive increasing attention. Biomaterials used as implants are divided into 4 groups: metallic, composite, polymeric and ceramic biomaterials. Due to higher strength and hardness and better biocompatibility, metallic biomaterials are used much more in biomedical applications. An alloy Ti-6Al-4V, discovered in the United States in 1954, thanks to its optimal biomechanical properties, became the standard metallic biomaterial in dental implantology. Titanium and its alloys are often described in the literature as "dental metals of the future". This is supported by the large number of new titanium alloys that are being discovered every day. Namely, titanium and its alloys as biomedical materials can improve the quality of human life and life expectancy and therefore in the last few decades have become increasingly used in dentistry and orthopedics [1-4].

1.1. The goal of the paper

With the development of new materials, the methods of their metallographic preparation are being developed or modified in order to obtain as accurate and reliable results as possible using various methods of determining their characteristics. Recently, more and more emphasis has been placed on the economy of the procedure, i.e. on saving resources. Therefore, this paper will study the influence of metallographic preparation of alloys, i.e. grinding and polishing, on the measured hardness values of experimental titanium alloy $Ti_{80}Cr_{15}Co_5$ designed for use in biomedicine [3], in order to determine the optimal conditions for its metallographic preparation. This will be assessed using a numerical analysis of the measured hardness values measurement results. For this purpose, by cutting the alloy into 12 equal parts, samples will be obtained which will be inserted into the epoxy mass, ground and polished. After grinding using various parameters, the surface condition will be recorded and the hardness of all samples measured by the Vickers method. Hardness will be measured on all samples also after polishing. From the obtained results, the influence of metallographic preparation on the measured hardness values of the experimental titanium alloy will be concluded.

2. Prerequisites and means for solving the problem

2.1. Preparation of samples

In order to determine the influence of metallographic preparation on the measured hardness values of the alloy $Ti_{80}Cr_{15}Co_5$ using a numerical analysis, the combination of parameters shown in Tab. 1 was selected. The rotation speed of the grinding wheel was kept constant at 200 rpm.

Table 1. *Grinding parameters of titanium alloy*

Force / time	3 min	6 min	9 min
5 N	sample 1	sample 5	sample 9
10 N	sample 2	sample 6	sample 10
15 N	sample 3	sample 7	sample 11
20 N	sample 4	sample 8	sample 12

In order to obtain the appropriate number of samples for metallographic preparation with different parameters according to Tab. 1, it was necessary to obtain twelve approximately equal parts of titanium alloy. Therefore, the cutting was performed on a JET HVBS-56M.

After cutting, the samples were inserted into the polymer mass. The samples were placed in a mold each separately and overflowed with a viscous two-component polymer resin CEM1000, a mixture of polymer in powder form and liquid catalyst.

Grinding and polishing of the samples were performed by standard procedure. Titanium alloy samples were grinded with a force of 5 N, 10 N, 15 N or 20 N for 3, 6 or 9 minutes, i.e. one of 12 possible combinations was applied to each sample. Grinding was performed with sandpapers of different grain sizes, from the coarsest (120) to the finest (1200). The grinding machine speed was set to 250 rotations per minute. During grinding the samples were constantly cooled with water to prevent possible changes in the microstructure due to the action of heat generated by friction between the grit paper and the surface of the metal sample and to remove / wash impurities and grinding products.

2.2. Light microscopy

A light microscope was used to obtain information about the appearance of the surface and phenomena in the samples, such as inclusions, porosity, cracks and others. For that purpose, samples that were metallographically prepared were used.

In this paper the samples were observed at 200 times magnification using an Olympus GX 51 light microscope. The surfaces of the experimental alloy samples after different conditions of metallographic preparation were recorded using an Olympus DP 70 digital camera connected to the said light microscope.

2.3. Hardness measurement

Hardness is a mechanical property that represents the resistance of a material to the penetration of another, harder material into its surface or structure. Hardness is affected by the chemical composition of the material, mechanical and heat treatment. Further, hardness is closely related to the yield strength, modulus of elasticity, fracture strength, tensile strength and toughness [5,6].

In this paper the hardness of the experimental alloy samples was measured by the Vickers method on a Mitutoyo hardness testing machine with different indentation forces: 50 N, 100 N and 300 N. Hardness measurements with each indentation force were performed 3 or 5 times at random spots on the polished surface. After that, all samples were polished again on a microfiber fabric (so-called felt) to a mirror surface (without scratches) at a speed of 200 rpm with occasional addition of aqueous suspension of Al_2O_3. The polishing time was 10-15 minutes. The hardness of HV5, HV10 and HV30 was also measured on the polished samples. The indentation time was 10 s, and the magnification of the microscope for the imprint diagonals measurement was 200 x.

2.4. Statistical analysis of hardness measurement results

In this paper 12 samples of titanium alloy were examined. All samples were grinded with four different grinding forces (5 N, 10 N, 15 N, 20 N), during three different times: 3 min, 6 min and 9 min.

Hardness was measured by the Vickers method using indentation forces of 50 N, 100 N and 300 N. Each measurement was repeated for 3-5 times and the average values were calculated. Values that deviated from the average by more than one standard deviation were excluded. After measuring the hardness with all the chosen indentation forces the samples were polished, and then all measurements were repeated. In repeated measurements, besides indentation forces of 50 N, 100 N, 300 N, a force of 500 N was also used to check the dependence of the measured hardness value on the indentation force obtained before polishing.

3. Results and discussion

3.1. Light microscopy results

Images of the surface of the samples after grinding in different conditions are shown in Fig. 1.

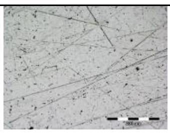
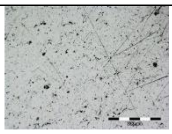
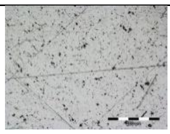
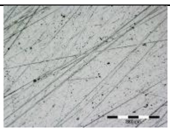
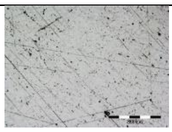
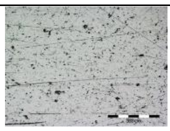
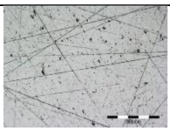
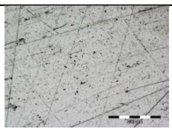
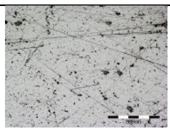
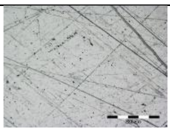
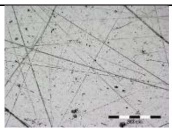
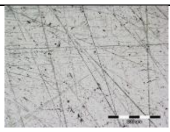
Time / Force	3 min	6 min	9 min
5 N			
10 N			
15 N			
20 N			

Fig. 1. *The condition of the surface of the samples after grinding by applying different forces for different times*

They show that grinding with the greatest force (20 N) results in the roughest and most numerous cuts / scratches, which become thinner with longer grinding. In the images shown it can be noticed that a longer grinding time leads to the smoothing of deeper cuts and a significant reduction in their number. When comparing the images taken after grinding for 3 and 6 minutes, a significant difference in the number and roughness or fineness of the scratches can be noticed. However, this difference after 6 and 9 minutes of grinding is not so noticeable. From the economic point of view, it follows from the above that the optimal grinding conditions would be the mean grinding time (6 min) and the mean grinding force (10 - 15 N).

3.2. Hardness measurement results

Tab. 2 shows the measurements before polishing while Tab. 3 shows results of measurement after polishing. Excessive deviations are marked in red and these measurements are not included in the calculation of the mean hardness value, which is indicated by the bold font for each sample and for each indentation force.

Table 2. *Hardness measurement results after grinding*

sam-ple	grind. force (N)	grind. time (min)	$HV5$	deviation (sigma)	$\overline{HV5}$	$HV10$	$\overline{HV10}$	$HV30$	$\overline{HV30}$
1	5	3	346,9	0,878	**328,4**	309,2	**313,6**	303,2	**307,6**
			322,1	-0,721		315,9		311,2	
			352,8	1,258		315,6		308,4	
			318,4	-0,959					
			326,2	-0,456					
2	10	3	317		**315,0**	311,3	**305,9**	313,9	**310,8**
			314,7			304,7		310	
			313,4			301,6		308,6	
3	15	3	315,4	-0,085	**312,6**	306,6	**309,2**	317,8	**315,0**
			330,9	1,451		312,2		311	
			320,9	0,460		308,8		316,3	
			306,4	-0,977					
			307,7	-0,849					
4	20	3	317		**315,5**	302,2	**305,5**	307,5	**302,2**
			314,2			301,6		300,4	
			315,3			312,6		298,8	
5	5	6	304,2		**308,4**	321,6	**321,1**	312,5	**315,1**
			311,6			318,1		315,1	
			309,5			323,7		317,7	
6	10	6	322,1	0,964	**315,5**	304,8	**306,6**	310	**312,5**
			320,8	0,839		301,5		309,2	
			298,3	-1,316		313,5		318,4	
			314,8	0,264					
			304,2	-0,751					
7	15	6	311,8	-1,384	**311,3**	320,6	**316,2**	307,7	**304,6**
			302,4	-1,990		317,2		300,9	
			324,3	-0,579		310,9		305,1	
			319,7	-0,875					
			293,6	-2,557					
8	20	6	321,6	0,463	**297,0**	309,6	**309,3**	304,6	**307,6**
			295,7	-3,148		306,7		314,2	
			275,1	-6,019		311,6		304	
			297,7	-2,869					
			297,6	-2,883					
9	5	9	315,6	0,384	**309,1**	316,6	**310,1**	313,8	**312,3**
			311,1	-0,348		306,9		312	
			298,5	-2,397		306,9		311,1	
			309,3	-0,641					
			306,8	-1,047					
10	10	9	299,7	-1,921	**309,5**	312,3	**311,7**	302,7	**306,6**
			312,3	-0,036		310,2		312	
			314,2	0,248		312,5		305,1	
			308,2	-0,649					
			307,9	-0,694					
11	15	9	316,3		**310,9**	312,3	**307,8**	311,9	**307,7**
			314,3			303,2		307,3	
			302			307,8		304	
12	20	9	309,5		**309,6**	314,2	**311,3**	311,8	**309,1**
			309			306		308,3	
			310,4			313,7		307,1	

In general, the results in Tab. 2 show relatively similar values of the measured hardness under all conditions of metallographic preparation. However, considering the grinding parameters (time and force) it can be seen that the grinding time has a more significant role, because after grinding the samples for 9 minutes almost identical hardness values were measured, while after grinding for 3 and 6 minutes the measured values show larger deviations.

Table 3. *Hardness measurement results after polishing*

sam-ple	grind. force (N)	grind. time (min)	$HV5$	deviation (sigma)	$\overline{HV5}$	$HV10$	$\overline{HV10}$	$HV30$	$\overline{HV30}$
1	5	3	322,4		**319,7**	314,1	**319,1**	310,2	**310,5**
			321,2			322,1		309,5	
			315,5			321,2		311,9	
2	10	3	346,2	1,292	**315,8**	309,6	**304,0**	309,8	**306,2**
			335,1	0,790		306		301,2	
			310,4	-0,325		296,4		307,6	
			294,4	-1,048					
			301,9	-0,709					
3	15	3	302,1		**311,9**	307,6	**304,2**	310,4	**309,9**
			316,1			303,4		308,9	
			317,6			301,5		310,4	
4	20	3	315,9		**321,6**	301,6	**299,8**	310,9	**305,5**
			326,7			296,7		306,6	
			323,1			301,2		299,1	
			320,8						
5	5	6	331,6	1,492	**309,9**	310,2	**311,3**	317,6	**312,3**
			310,1	-0,482		314,8		309,2	
			308,5	-0,629		308,9		310,2	
			311,2	-0,381					
6	10	6	307,7		**308,2**	303,8	**308,1**	311,3	**311,2**
			300,3			312,2		316,2	
			316,5			308,4		306,1	
7	15	6	318,9	-0,546	**314,6**	317,6	**308,0**	313,1	**311,5**
			324,8	-0,112		302,7		313,8	
			312	-1,054		303,6		307,7	
			313	-0,981					
8	20	6	304,9		**297,5**	309,2	**305,0**	305,7	**304,4**
			291,2			294,7		305,4	
			296,3			311,2		302,2	
9	5	9	329,1	2,301	**329,6**	322,9	**318,5**	307,8	**305,1**
			340,3	3,703		316,3		303,9	
			343,2	4,066		316,3		303,5	
			319,5	1,099					
			316,3	0,699					
10	10	9	299,6		**306,0**	313,8	**316,7**	302,3	**310,1**
			307			316,9		312,1	
			311,4			319,5		316	
11	15	9	325,5	1,164	**307,8**	297,2	**303,0**	319,9	**309,6**
			325,3	1,144		304		303,8	
			305,2	-0,859		307,7		305	
			306,3	-0,750					
			311,9	-0,191					
12	20	9	322,4	0,694	**315,0**	309,3	**308,2**	308,1	**311,0**
			327,7	1,200		306,7		316,1	
			312,1	-0,289		308,7		308,8	
			310,5	-0,441					

3.3. Statistical analysis of hardness measurement results

After measuring the hardness of all samples with different indentation forces, statistical analysis of the results was performed. First, the mean hardness value was calculated and also the standard deviation from the mean value, for all 12 samples together, depending on the indentation force, before and after polishing. The obtained results are shown in Tab. 4:

Table 4. *Comparison of calculated mean hardness values and standard deviations*

HV		HV5	HV10	HV30	HV50
Before polishing	mean	311,9	310,7	309,3	-
	standard deviation	7,2	4,6	4,0	-
After polishing	mean	313,1	308,8	309,0	306,9
	standard deviation	8,3	6,4	2,8	4,7

The results given in this table show that the differences in the measured hardness before and after polishing are much smaller than the standard deviation of the measurement series, which means that the polishing process does not significantly affect the measured hardness values of the experimental titanium alloy.

Furthermore, the standard deviations are slightly higher for the indentation force of 50 N (although measurements with excessive deviation have already been omitted), which means that the indentation force of 50 N is too small to measure the hardness of the experimental titanium alloy, i.e. measurements with this force result in excessive statistical errors. This is quantified by taking the first three measurements for each sample (regardless of deviations) and calculating the relative measurement error (RME) according to the expression:

$$(1) \quad RME = \frac{maximum\ \ hardness\ \ - minimum\ \ hardness}{mean\ \ hardness\ \ value}.$$

This term was used due to the fact that the standard deviation is not reliable information because it was calculated from a series of only three measurements. The mean value for RME was then calculated for all 12 samples and the results are shown in Tab. 5.

Table 5. *Relative measurement error*

Indentation force		50 N	100 N	300 N	500 N
Before polishing	RME	0,053	0,025	0,023	-
After polishing	RME	0,051	0,034	0,033	0,032

The results shown in Tab. 5 show that the RME is much higher for a force of 50 N than for other applied forces. For this reason, it is recommended to use an indentation force of at least 100 N to measure the hardness of the experimental titanium alloy.

From the results obtained by measuring the hardness, a decrease in the measured hardness (HV) with an increase in the indentation force (F) is also observed. For the values obtained after grinding (before polishing) the following formula is obtained:

$$(2) \quad HV = 309.105 + 4.9404 \cdot e^{-0.011F},$$

with the R^2 value equal to 1, which means a perfect match. The quality of this model was checked by measuring the hardness of all samples after polishing with an additional indentation force of 500 N and the following formula was obtained:

$$(3) \quad HV = 306.68 + 7.2185 \cdot e^{-0.006F},$$

with the R^2 value equal to 0.8306, so still a good match, which is clearly shown by the graph shown in Fig. 2:

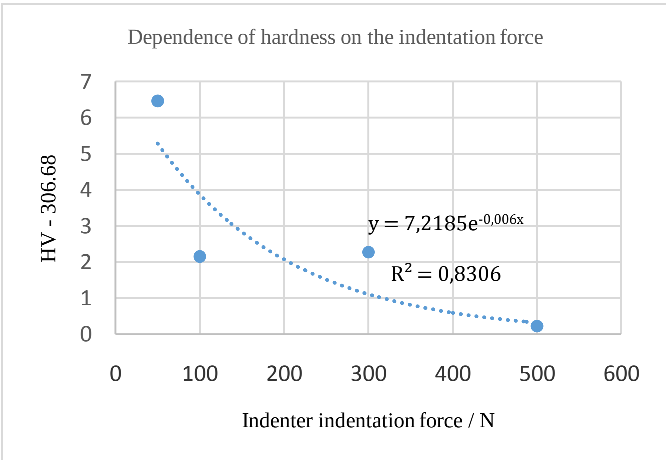


Fig. 2. *Dependence of hardness measured after polishing on the indentation force*

The dependence of the measured hardness on the grinding parameters was also determined. However, the standard deviation for only three measurements is mostly in the range of 3 to 7 HV, so statistically significant deviations from the mean values are only those greater than 10 HV. Statistically significant deviations are considered to be those that deviate from the mean by at least two standard deviations. There were very few such large deviations in these measurements, so unfortunately no clear conclusion can be drawn in this direction. Therefore, it is recommended that a higher number of measurements be performed during the hardness test, with fewer variable parameters.

However, it can be observed that the hardness decreases with the grinding force because such a trend is obtained both before and after polishing, as shown in Fig. 3 and 4 (a mean hardness value of measurements for all grinding times is considered here).

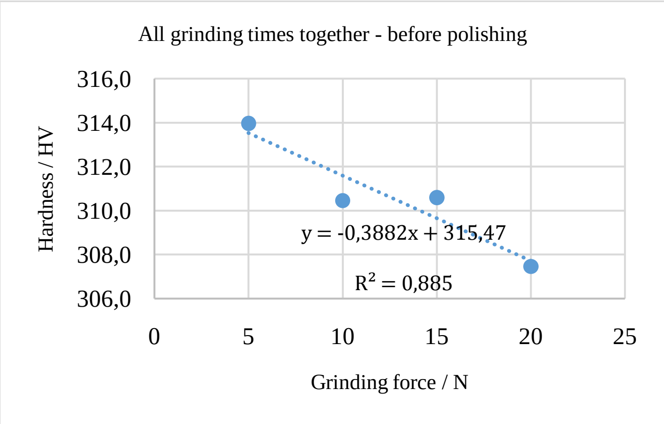


Fig. 3. *The trend of decreasing hardness with increasing grinding force - before polishing*

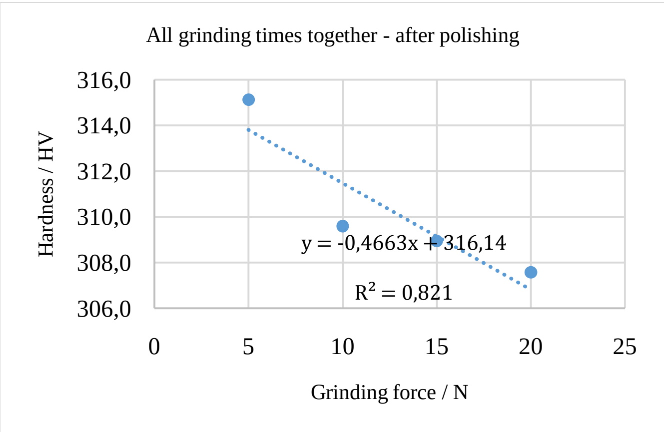


Fig. 4. *The trend of decreasing hardness with increasing grinding force - after polishing*

4. Conclusion

In this paper we measured the hardness of 12 samples of experimental titanium alloy $Ti_{80}Cr_{15}Co_5$ by the Vickers method with different indentation forces (50 N, 100 N, 300 N) after grinding for 3, 6 and 9 minutes with grinding forces of 5, 10, 15 and 20 N, in order to determine the influence of metallographic preparation on the measured hardness values of the alloy. The measurements are done both before and after polishing.

Based on the results obtained in this paper, the following conclusions can be drawn:

- After grinding the samples for the longest time (9 min) almost all samples have no cuts / scratches or are they slightly visible. The exception is sample 12, which was ground with the greatest force, and this obviously led to significant damage to the surface, which was not smoothed even after 9 minutes of grinding.
- The measured hardness values are very similar (uniform) which can be explained by the homogeneous microstructure of the experimental alloy.
- The measured hardness values decrease as the surface of the samples becomes finer.
- The values of hardness measured during indentation with higher forces do not depend on the surface condition, i.e. on the metallographic preparation of the samples.
- The hardness values measured during indentation with lower forces increase with increasing surface roughness of the samples.
- The measured values of the hardness of the samples of the experimental titanium alloy decrease with increasing indentation force.
- The measured hardness values decrease with increasing applied grinding force in the metallographic preparation of samples.
- The polishing process does not have a significant effect on the measured hardness values of the experimental titanium alloy.
- Indentation force of 50 N is not recommended since the standard deviations are slightly higher than with higher indentation forces.
- From the aspect of economy, in order to save resources (grinding material, water, electricity), it is enough to use the average grinding force (10-15 N) for the average time (6 min), after which the relevant hardness values are obtained.

5. References

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