

This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivs 3.0 Unported License. To view a copy of this license, visit http://creativecommons.org/licenses/by-nc-nd/3.0/ or send a letter to Creative Commons, 444 Castro Street, Suite 900, Mountain View, California, 94041, USA.

PROPERTIES AND STRUCTURE OF OXIDE LAYERS ON A THIN COATING OF A TITANIUM-NIOBIUM ALLOY

Jan Krčil^{1,*}, Jana Sobotová¹

¹ Institute of Materials Engineering, Faculty of Mechanical Engineering, Czech Technical University in Prague, Karlovo náměstí 13, 121 35 Prague 2, Czech Republic

*corresponding author: e-mail: jan.krcil@fs.cvut.cz

Resume

Present work discusses issues of growth and characterization of a thin oxide layer formed on the surface of a titanium-niobium alloy. An oxide layer on the surface of titanium alloys introduces a corrosion resistance and also a bio-compatibility, which is required for a medical application. Although this oxide layer is a result of a spontaneous passivation, for the practical applications it is necessary to control the growth of oxides. In this work the oxide layer was formed on the PVD coating from Ti39Nb alloy which was sputtered on three different base materials: CP Ti grade 2, stainless steel AISI 316LVM and titanium alloy Ti–6Al–4V ELI. The oxide layer was created by a thermal oxidation at 600 °C for three different oxidation periods: 1, 4 and 8 hours. After the oxidation process the influence of oxidation characteristics and base materials on the thickness and properties of oxide layer was studied. There was observed a change of color and surface roughness. The oxide layer surface as well as the layer thickness was observed by SEM. The influence of the substrate material under the coating on the oxide layer should be more investigated in the future.

Article info

Article history: Received 01 July 2015 Accepted 12 October 2015 Online 17 December 2015

Keywords: Biomaterials; Oxide layer; Titanium; PVD coating; Niobium; Thermal oxidation.

Available online: http://fstroj.uniza.sk/journal-mi/PDF/2015/17-2015.pdf

ISSN 1335-0803 (print version) ISSN 1338-6174 (online version)

1. Introduction

The need for better bio-materials used for medical application (implants and surgical tools) growths significantly with prolonging of active human life and with the increase of physical activity. This leads to ways of research heading towards finding new materials, or improving old materials, which would perform better in the areas of bio-compatibility (or bio-activity), mechanical properties, corrosion resistance and others. The aim is to find a material which would interage as a natural part of body, would be able to bear stress and which would be economically beneficial. One of the possible ways of development is the usage of coating from highly bio-compatible material on the base material from material with acceptable mechanical properties [1, 2].

This work deals with creation, kinetic of growth and with properties of oxide layer formed on the thin coating from titanium alloy. The work aims to be a starting point for further investigation. Because the problematic of thermal oxidation of materials with thin PVD coating was not fully researched. The results bring knowledge how to improve surface treatment of materials applied in bio-medicine.

1.1 Biomaterials

The stainless austenitic steel AISI 316LVM is a material commonly used for orthopedic implants and is widely chosen because of its low price, easy production and less complicated welding in comparison to titanium, titanium alloys and alloys on Cr-Mo basis, while the properties for biomedical application are maintained [1].

Pure titanium, known as CP titanium (where CP stands for Commercially Pure) is another material widely used in many biomedical applications, e.g. cranial plates, maxillofacial reconstruction, dental implants, bone fracture fixations and prosthetic joints [2]. The most common among CP Ti materials are four grades, CP Ti grade 1 (99.5 wt. % Ti), grade 2 (99.3 wt. % Ti), grade 3 (99.3 wt. % Ti) and grade 4 (99.0 wt. % Ti). Although each material contains slightly different levels of N, Fe, and O, C is specified <0.10 wt. % and H is also specified <0.015 wt. % [2, 3].

Ti–6Al–4V alloy belongs to titanium $\alpha + \beta$ alloys and because of the combination of high resistance against corrosion, good chemical and mechanical properties it is one of the most used alloys in bio-medicine. Although some of the studies [3, 4] indicate that there is a possibility of releasing ions of Al and V as a result of a longterm contact with the human body. These ions may cause the Alzheimer's disease or osteomalacia [2, 3, 5, 6].

Due to the possible toxicity of Ti–6Al–4V alloys and further effort to improve the properties of bio-materials, new materials are being investigated. One of the ways to use Ti alloys without Al and V addition. The titanium β alloys, stabilized by Nb, are one of the possibilities; they are achieving the suitability in mechanical properties as in corrosion resistance and biocompatibility [1, 5 – 8].

1.2 Oxide layer

The corrosion resistance of titanium and its alloys is caused by a thin oxide layer which is forming itself on the surface of material. The forming of this layer is a result of high reactivity of titanium and the layer growth is initiated in just micro-seconds after the exposure of Ti material to the atmosphere or moisture [2].

The growth rate of oxide layer may be increased by changing of external conditions – exposing the material to stronger oxidizing environment, e.g. heating on the higher temperatures, exposure to stronger oxidants $(HNO_3, CrO_3 \text{ etc.})$, or anodic oxidation [9, 10].

The resulting oxide layer is composed of three oxides: TiO₂, Ti₂O₃ and TiO. Among these, the titanium dioxide (TiO₂) is the most common and the most thermodynamically stable, it is the primary oxide from which the oxide layer on Ti and Ti alloys is consisted. TiO2 occurs in nature in three modifications (anatase, brookite and rutile); it is possible to obtain other modifications synthetically and due to high pressure. The three modifications have the same chemical composition, they differ in the configuration of their atoms. This different structure is the reason for the different properties; the rutile phase is stable in every temperature (as oppose to anatase and brookit which are unstable), and as the author [2] notes, is the most suitable for the human body [2, 11-13].

The layer is primarily formed by titanium oxides, but in the composition may appear more complex oxides (e.g. Al_2TiO_5 on Ti-6Al-4V, Nb_2O_5 and NbO_2 on TiNb, or NiTiO_3 on NiTi). In other cases the titanium oxides doped with small amount of other elements (elements from the substrate material or the oxidation atmosphere). These small doses may then affect the properties of oxide layer (e.g. TiO_2 on TiNb alloys may be doped with small amount of Nb, this results in better bio-compatibility) [2, 12, 14].

2. Experiment

Three specimens of CP Ti grade-2, Ti– 6Al–4V ELI alloy and stainless steel AISI 316LVM in the form of cyllindric bars, were used in this study (see Table 1 for chemical composition of materials).

The bars were cut into the specimens with diameter 15 mm and thickness 8 mm. Flat planes of specimens were ground and polished to obtain a surface with a mirror look. The PVD coating was then created on the polished specimen surfaces (see Table 2 for PVD coating parameters) in the Flexicoat 850, Hauzer. For the coating was used target made of alloy Ti39Nb. Specimens with PVD coating were subject to thermal oxidation.

2.1 Experiment characterization

The parameters of the oxidation were chosen according to the literature [2, 7, 8, 15]. The sample was subjected to the oxidation at 600 °C in a furnace LAC LH 30/13 with regulator MT825-A at the presence of air on atmospheric pressure for 3 distinct periods: 1, 4 and 8 hours.

The furnace heating started at room temperature and continued gradually with speed of 7.5 °C/min up to 600 °C. After the oxidation period elapsed, the heating was switched off and the specimens were left to cool down to the room temperature. For the details about the oxidation process (speed of heating and cooling) see Fig. 1. This form of oxidation process was expected to provide oxide layers with good adhesion to substrate material.

Table 1

Chemical compositions of CP Ti grade 2, stainless steel AISI 316LVM and Ti-6Al-4V ELI. Chemical composition of CP Ti grade 2 (wt %) (maximal amounts)

	Chemical composition of C1 11 grade 2 (wt. 76) (maximal amounts)									
0	Ν	С	Н	Fe	Al	\mathbf{V}	Ni	Mo	others	Ti
0.25	0.0300	0.0800	0.0125	0.3000						balance
Chemical composition of stainless steel AISI 316LVM (wt. %) (maximal amounts)										
С	Si	Mn	Р	S	Cr	Mo	Ni	Cu	Ν	PRE
0.0220	0.3500	1.7500	0.0210	0.0010	17.1800	2.7100	14.1000	0.0600	0.0940	26.1230
Chemical composition of alloy Ti-6Al-4V ELI (wt. %) (maximal amounts)										
0	Ν	С	Н	Fe	Al	V	Ni	Mo	others	Ti
0.1300	0.0500	0.0800	0.0120	0.2500	6.5000	4.5000				balance

The parameters of PVD coating.

Table 2

Parameter Tempe	erature (°C)	Current (A)	Sputtering power (kW)	Pressure (Pa)	Thickness (µm)
Value	250	5	4	200	1.0-1.5



(full colour version available online)

After the removal of specimens from the furnace was on their surface observed a change of color and the roughness of surfaces was measured. Then the surface of the specimens was observed and the thickness of the oxide layer was measured by the scanning electron microscope JEOL JSM 7600F equipped with an energydispersive X-ray analysis system (EDS).

3. Results and discussion

3.1 Surface color

The observation of the surfaces of oxidized specimens has shown the change of color. It can be seen that the color is changing with the increase of the oxidation period.

For the shorter periods of time, the color was dark blue. For the specimens from AISI 316LVM with TiNb coating (see Fig. 2) can be seen that the color changes into the purple with gold accent (4 hours) and silver accent (8 hours) with longer oxidation. For the titanium based substrate materials with Ti39Nb coating (see Fig. 3 and 4) the blue color changed into light blue/green with silver accent. All these observation of the surface colors coincides with observations described in [2]. The change of color indicates the creation of oxide layer, and further change of color with prolonging oxidation period indicates change in the thickness of oxide layer.

3.2 Surface roughness

As it is described in literature [2], the growth of oxides on the surface of specimens leads to increase of the roughness of the surface: the roughness is increasing with the thickness of layer and the size of oxides. The change of surface roughness, as well as the change of surface color, indicates the existence of oxide layer. In this work the objective was to measure the roughness of oxidized specimens and observe the changes of the surface roughness based on (a) the oxidation conditions and on (b) the substrate material. The surface roughness in Table 3. measurement is summarized The results shown that oxidized specimens have higher surface roughness which increases with longer oxidation period. Observed results agree with outcomes mentioned in [2].



Fig. 2. Surface color on 316LVM+Ti39Nb; oxidation 600 °C; a) 1 hour; b) 4 hours; c) 8 hours. (full colour version available online)



Fig. 3. Surface color on Ti grade-2+Ti39Nb; oxidation 600 °C; a) 1 hour; b) 4 hours; c) 8 hours. (full colour version available online)



Fig. 4. Surface color on Ti–6Al–4V ELI+Ti39Nb; oxidation 600 °C; a) 1 hour; b) 4 hours; c) 8 hours. (full colour version available online)

Table 3

Surface roughness of specimens; Ti g 2, 316LVM and Ti-6Al-4V ELI with Ti39Nb coatings.

Test condition	Ra (µm) (average value)						
Test condition	CP Ti grade 2	Steel AISI 316LVM	Ti–6Al–4V ELI				
Not-oxidized	0.030	0.010	0.020				
Oxidized: 600 °C, 1 hour	0.051	0.020	0.060				
Oxidized: 600 °C, 4 hours	0.060	0.026	0.074				
Oxidized: 600 °C, 8 hours	0.086	0.053	0.083				



Fig. 5. Change of surface roughness with prolonging oxidation period; CP Ti grade 2, 316LVM and Ti–6Al–4V ELI with Ti39Nb coatings. (full colour version available online)

The results were put into a chart (see Fig. 5) to visualize the evolution of surface roughness with increasing oxidation time.

The growth of roughness was observed for all the substrate materials with Ti39Nb coating. After the 8-hour oxidation the surface roughness was

approximately three times higher than on the untreated specimens for all substrate materials. To compare the materials, we have calculated a linear regression of each substrate. It can be seen that the increase rate is roughly the same for all substrate materials with Ti39Nb coating. The only difference is due to the initial states of the specimen surfaces. Even though all of specimens were prepared in the same manner, difference was predetermined by the substrate materials themselves.

3.3 Surface structure and layer thickness

The surface of specimens with a substrate material 316LVM (see the left micrograph in the Fig. 6) indicates a presence of crystal oxides (marked in Fig. 6a) even after a short oxidation period. The size and mount of oxides rapidly increases with the oxidation period. This coincides with the measurement of oxide layer thickness (right micrograph in Fig. 6). In consent with growth of crystals was detected measurable oxide layer (see the right side of Fig. 6).



Fig. 6. SEM micrograph of specimen surface and cross-section detail view of specimen with oxide layer; 316LVM+Ti39Nb; oxid. at 600 °C, for a) 1; b) 4; c) 8 hours.

In the case of 4-hour oxidation (Fig. 6b) was observed a 120 nm tick oxide layer with worse contrast. After the 8-hour oxidation (Fig. 6c) a clearly visible 240 nm layer was detected.

The surface of specimens with substrate material CP Ti grade 2 (see the left micrograph in Fig. 7) exposed to a short-term oxidation indicates a presence of crystal oxides (marked in Fig. 7a). With longer oxidation period the size and amount of crystals increased but not as much as in the previous case. The measurable oxide layer was also detected (see the right side micrograph in Fig. 7). In the case of 4-hour oxidation (see Fig. 7b) the oxide layer was rather thin (54 nm) and poorly distinguishable. After the 8-hour oxidation (see Fig. 7c) was detected a compact and strong oxide layer (290 nm). However, its contrast is lower than in the case of specimens from 316LVM.



Fig. 7. SEM micrograph of specimen surface and cross-section detail view of specimen with oxide layer; CP Ti grade 2+Ti39Nb; oxid. at 600 °C, for a) 1; b) 4; c) 8 hours.

We have found just a small amount of oxide crystals on the surface of specimens with substrate material Ti–6Al–4V ELI (see the left micrograph in Fig. 8). The amount and the size of crystals was increasing with oxidation period length but not as much as it was observed on previous two groups of specimens. We also have not detected any measurable oxide layer on any of these specimens (see the right micrograph in Fig.8).

A comparison of the micrographs

of surface and the measurements of the coating and oxide layer showed that there is a visible relationship. The growth of oxides (i.e. both the size as well as the amount of the oxide particles) on the surface of coating indicated (as well as the change of the color and roughness) the increase of thickness of the oxide layer. Measurements of the size, the amount and the placement of oxides on the specimen surface were not realized. It remains as an objective for later works.



Fig. 8. SEM micrograph of specimen surface and cross-section detail view of specimen; Ti–6Al–4V *ELI+Ti39Nb; oxid. at 600 °C, for a) 1; b) 4; c) 8 hours.*

It was observed that the oxide layer does not depend only on the coating; the growth of the oxides was also affected by the substrate material. The best results were obtained on the specimens with the substrate material from stainless steel 316LVM. The oxide layer is compact, well visible and measurable as well as the oxide crystals on the surface. However, we have not observed any measurable oxide layer on the surface of specimens with substrate material from Ti-6Al-4V ELI only isolated crystals are present on the surface (though their size exceeds that of all other specimens). The influence of the substrate material under the coating on the oxide layer should be more investigated in the future.

On the specimen where the oxide layer was the best contrast (316LVM with Ti39Nb coating oxidized for 8 hours) the EDS analysis was performed (see Fig. 9). It is a line EDS analysis allowing an approximate identification of elements. The blue line stands for oxygen, red for titanium, yellow for niobium, turquoise for iron and others are backing elements (more detailed results see on the Fig. 10). Oxygen signalizes the oxide layer. Ti and Nb are present in the oxide layer as well as in the PVD coating. Fe, Cr and Ni are in results present due substrate material. The higher amount of silicon is in results present due to the preparation of the specimen (silica gel was used during polishing).

It can be seen, that in the substrate material Fe prevails, whereas Ti and Nb form the majority in the coating and oxygen prevails in the surface layer. As it has been mentioned above, these results are not exact and serve only as the approximate verification of the oxide presence in the newly formed layers. In case of any other experiments it will be more appropriate to use other methods of EDS analysis and X-ray diffraction to analysis the phases of the oxides. These methods will not only provide more exact data, but it will be possible to distinguish and define various oxides and their structures.



Fig. 9. SEM EDS line scan results of cross-section of the studied specimen 316LVM with Ti39Nb coating, oxidation: at 600 °C, for 8 hours. (full colour version available online)



Fig. 10. SEM EDS line scan results of cross-section of the studied specimen 316LVM with Ti39Nb coating, oxidation: at 600 °C, for 8 hours; a) Oxygen; b) Titanium; c) Niobium; d) Ferrum; e) Chromium; f) Nickel; g) Silicon. (full colour version available online)

4. Conclusions

- A thermal oxidation was preceded on a thin coating consisting of an alloy Ti39Nb which was sputtered on the surface of CP Ti grade 2, stainless steel AISI 316LVM and alloy Ti-6Al-4V ELI.
- A change of color was observed on the surfaces of oxidized specimens. This indicated a formation and existence of oxide layer of variable thickness.

157

• All of the oxidized specimens were showing the influence of oxidizing conditions on the surface roughness.

- Different growth rates of surface oxides throughout the oxidation period, depending upon the substrate material, were noted.
- There is no measurable oxide layer on the coating from alloy Ti39Nb on the specimens from the alloy Ti-6Al-4V ELI.
- In the case of specimens from CP Ti grade 2 and stainless steel AISI 316LVM were measured an oxide layer of variable thickness on the coating from alloy Ti39Nb, after the oxidation period of 4 and 8 hours.
- The thickest oxide layer was noted on the specimen with the substrate material CP Ti grade 2 with a coating from alloy Ti39Nb, after the oxidation during 600 °C and for the period of 8 hours.
- The relationship between growth of the oxides on the surface and the measured thickness of oxide layer was noted.
- In the next study, the characterization and the analysis of the oxide layers and their structure is considered to be done. The idea is to use the X-ray diffraction to identify the compounds and structured in the oxide layer.

Acknowledgements

This work was supported by the Ministry of Education, Youth and Sport of the Czech Republic, programme NPU1, project No LO1207.

Note

Shorter and less complex version of this article was presented at the 25th Heat Treatment Days conference in Jihlava.

References

- [1] U. Kamachi Mudali, T.M. Sridhar, R. Baldev: Sadhana 28(3) (2003) 601-637.
- [2] O. Yoshiki: Bioscience and bioengineering of titanium materials, Elsevier, London 2013.
- [3] J. Janovec, J. Cejp, J. Steidl: Perspektivní materiály (*Perspective materials*), Publisher Czech Technical University in Prague, Prague 2008 (*in Czech*).
- [4] G. Manivasagam, D. Dhinasekaran, A. Rajamanickam: Recent Patents on Corrosion Science 2(1) (2010) 40-54.
- [5] F. Zhang, E. Burkel: In: Biomedical Engineering, Trends in Materials Science, Ed.: A. Laskovski, 2010, pp. 203-224.
- [6] I. Jirka, M. Vandrovcová, O. Frank, Z. Tolde, J. Plšek, T. Luxbacher, L. Bačáková, V. Starý: Mater. Sci. Eng. C Mater. Biol. Appl. 33(3) (2013) 1636-1645.
- [7] A. Gutiérrez, M.F. López, J.A. Jiménez, C. Moranz, F. Paszti, A. Climent: Surf. Interface Anal. 36(8) (2004) 977-980.
- [8] G. Zorn, A. Lesman, I. Gotman: Surf. Coat. Technol. 201(3-4) (2006) 612-618.
- [9] Corrosion resistance of titanium, [online]. [cit. 2014-01-09]. Available from: http://www.timet.com/document/technicalmanual s/corrosion.pdf.
- [10] Z. Abdolldhi, M.A.A. Ziaee, A. Afshar: International Journal of Chemical and Biomolecular Engineering 2(1) (2009) 179-182.
- [11] H. Dong, T. Bell: Wear 238(2) (2000) 131-137.
- [12] J. Huiren, H. Mitsuji, L. Yun, I. Hitoshi: Scripta Mater. 46(9) (2002) 639-643.
- [13] M.D. Hamilton, A. Butt, S. Patel, C. Sukotjo, D. Royhman, C.G. Takoudis: Journal of Undergraduate Research 6(1) (2013) 16-19.
- [14] S.D. Sartale, A.A. Ansari, S.-J. Rezvani: Mater. Sci. Semicond. Process. 16(6) (2013) 2005-2012.
- [15] J. Krčil: Nanášení a charakterizace oxidových vrstev na titanu: Bachelor thesis (*Deposition and characterization of oxide layers on titanium*) Prague: Czech Technical University in Prague, 2012 (*in Czech*).



This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivs 3.0 Unported License. To view a copy of this license, visit http://creativecommons.org/licenses/by-nc-nd/3.0/ or send a letter to Creative Commons, 444 Castro Street, Suite 900, Mountain View, California, 94041, USA.

R. Štěpánek, L. Pantělejev: Changes in mechanical properties of as-cast magnesium alloy AZ91 after equal channel angular pressing

CHANGES IN MECHANICAL PROPERTIES OF AS-CAST MAGNESIUM ALLOY AZ91 AFTER EQUAL CHANNEL ANGULAR PRESSING

Roman Štěpánek^{1,*}, Libor Pantělejev¹

*corresponding author: e-mail: stepanek.r@fme.vutbr.cz

Resume

This paper deals with differences in mechanical characteristics of AZ91 magnesium alloy in the as-cast state and after the subsequent equal channel angular pressing (ECAP) and with measurement of local microhardness. According to the obtained results, the tensile properties increased by a factor of two due to the ECAP procedure. The proof stress, $\sigma_{0.2}$, increased to 160 MPa and the ultimate tensile strength, σ_{UTS} , to 321 MPa. The local microhardness of ECAPed alloy varied between 70 and 91 HV 0.025 due to microstructural bimodality.

Article info

Article history: Received 09 November 2015 Accepted 30 November 2015 Online 20 December 2015

Keywords: AZ91; ECAP; Mechanical properties.

ISSN 1335-0803 (print version)

ISSN 1338-6174 (online version)

Available online: http://fstroj.uniza.sk/journal-mi/PDF/2015/18-2015.pdf

1. Introduction

Magnesium and its alloys as one of the lightest structural material seem to be suitable for application in industries where weight reduction is required. In fact, the properties of magnesium alloys in their most common as-cast state are not sufficient for many advanced applications [1]. In many cases, the conventional extrusion of magnesium alloys leads to satisfactory properties. However, a further grain refinement is required for special applications.

One of the possible ways is processing via severe plastic deformation (SPD) methods. One of the most used processing method is equal channel angular pressing (ECAP). This method provides homogenous microstructure of processed alloy and whole process of grain refinement can be very well controlled [2, 3, 4].

Although ECAP provides further grain refinement compared to conventional methods (e.g. extrusion), mechanical properties enhancement is not always guaranteed. The final properties are dependent on various factors, such as the initial state of the material or type of the alloy [5, 6]. Resulting mechanical characteristic is usually attributed to specific texture in the particular material [6].

Even though ECAP leads to mostly homogenous microstructures, the final microstructure often exhibits certain degree of bimodality depending on the processing conditions and specific material [4, 7, 8]. This bimodality leads to significant differences between local material properties and causes differences in the local response to mechanical loading. It can be postulated that plastic deformation in larger grains is easier contrary to areas of smaller grains which are rather resistant to plastic deformation [9].

This paper deals with enhancement of the tensile properties of as-cast magnesium alloy AZ91 after subsequent ECAP and with differences between the overall and local microhardness HV 0.025 of the alloy in the ECAPed state.

¹⁶⁰

¹ Institute of Materials Science and Engineering, NETME centre, Faculty of Mechanical Engineering, Brno University of Technology, Technická 2, 616 69, Brno, Czech Republic

2. Material and experimental methods

AZ91 magnesium alloy with chemical composition given in Table 1 in as-cast state was used in this work. The evaluated alloy was processed by ECAP in Ufa State University laboratory, Russia. The material was prepared by six passes through ECAP die at 300 °C using B_C route, the angle between intersecting die channels was of 120°. Material after ECAP processwas machined into cylindrical billets with diameter of 15 mm and length of 100 mm. Metallographic analysis was performed using light optical microscope Zeiss Axio Observer Z1.m. Samples for tensile tests were machined from billets so their longitudinal axis was identical the ECAPed with billets axis. Dimensions of the gauge length of the cylindrical testing specimens were of \emptyset 6 mm \times 30 mm. The tensile tests were performed using Zwick Z250 testing machine at room temperature with loading speed 2 mm/min. Microhardness was measured using Leco LM hardness tester. Different loads 247AT for hardness measurement were used, 0.05 kg (HV 0.05) for measurement of overall microhardness and 0.025 kg (HV 0.025) for measurement of microhardness in individual grains. For microstructural analysis at higher magnification, determination of grain size (EBSD), chemical microanalysis of particles (EDS analysis) and fractographic analysis on specimens failed during the tensile test for both states the analytic system Zeiss Ultra Plus (SEM) was used.

3. Results

The microstructure of the investigated alloy in the as-cast state exhibited typical features of a cast material (in this case dendrites in Fig. 1a) with eutectics along grain boundaries of solid solution (Fig. 1b) with grain size of about 500 μ m. The bimodal microstructure after ECAP consisted of fine grains (usually less than 5 μ m in diameter) in areas rich in Mg₁₇Al₁₂ phase (Fig. 2a – dark areas) and of areas with coarse grains (more than 15 μ m in diameter) practically

without $Mg_{17}Al_{12}$ particles (Fig 2a – light areas, Fig. 2b – solid arrows). The average grains size determined by EBSD was 6.1 µm. The material in both states also contained relatively large amount of manganese-based particles (determined by EDS analysis) homogenously distributed through the whole volume of samples (Fig. 2b – dashed arrow).

Fig. 3 shows typical engineering stress-strain curves (tensile testing at room temperature) for the analyzed alloy, in the as-cast state, and after the ECAP process. The obtained results (average values determined from three tensile tests) are summarized in Table 2. The mechanical properties of the as-cast material, i.e. $\sigma_{0.2}$ proof stress and the ultimate tensile strength were 87 MPa and 168 MPa respectively. The elongation of the as-cast alloy reached 3.1%. The ECAPed alloy exhibited a significant increase of all the measured properties; $\sigma_{0,2}$ proof stress 160 ultimate tensile strength of MPa, of 321 MPa, and elongation of 15.6%. The reduction of area was 5.1% for as-cast alloy and 14.3% for ECAPed alloy.

The microhardness was 64 HV 0.05 for the as-cast alloy and 89 HV 0.05 for the material after ECAP process. The specific values for the corresponding state of the alloy were determined as an average value from linear indentation measurements, each consisting of fifty imprints spanning all different microstructural areas. The local microhardness of the ECAPed alloy was measured in selected areas with fine grain clusters and in the areas of coarse grains. The microhardness of the fine grain areas obtained from three individual measurements was of 91 HV 0.025, while the microhardness

of the coarse grains reached value of 74 HV 0.025. It is necessary to note that the area fraction of coarse grained regions was 20 % only.

Fracture surfaces of the samples failed during the tensile test were analyzed by means of SEM. In the case of the as-cast samples, the fracture surface consisted of transgranular, quasi-cleavage fracture in which the cleavage planes and tearing edges appeared (Fig. 4a). Observation using higher magnification has shown that the ductile dimples are tiny and shallow (Fig. 4b). The material after six ECAP passes exhibited more ductile fracture. The fracture surface consisted of tiny and shallow dimples of substantial larger amount in comparison with the as-cast state, while the presence of cleavage planes/facets was rather poor (Fig. 5).

Table	1
-------	---

Chemical compositions of the analyzed alloy in wt. %.							
A 701	Al	Zn	Mn	Si	Fe	others	Mg
AL91	8.700	0.650	0.250	0.006	0.003	0.002	balance



Fig. 1. Microstructure of the as-cast alloy, a) typical microstructure, b) detail of the eutectic. (full colour version available online)



Fig. 2. Microstructure of ECAPed alloy, a) longitudinal plane, b) detail of bimodal area – longitudinal plane. (full colour version available online)

						Table 2				
	Grain size and mechanical properties at room temperature (average values).									
State of material	Average grain size (μm)	Proof stress σ _{0.2} (MPa)	Ultimate tensile strength (MPa)	Elongation (%)	Reduction of area (%)	Microhardness (HV 0.05)				
as-cast	>500	87	168	3.1	5.1	64				
ECAPed	6.1	160	321	15.6	14.3	89				

Materials Engineering - Materiálové inžinierstvo 22 (2015) 160-165



Fig. 3. Engineering stress-strain curves of the samples tested at room temperature [9, 10].



Fig. 4. Fracture surface of as-cast specimen failed during the tensile test a) quasi-cleavage fracture micromechanism, b) detail of shallow dimples close to cleavage facets.



Fig. 5. Fracture surface of specimens in ECAP state failed in tensile test a) quasi-cleavage fracture micromechanism, b) detail of ductile fracture with tiny dimples.

4. Discussion

According to the results, processing the as-cast AZ91 magnesium alloy by ECAP leads to a significant improvement of mechanical properties. Both $\sigma_{0.2}$ proof stress and ultimate tensile strength increased twice by ECAP (from 87 MPa 160 MPa to and 168 MPa to 321 MPa respectively). from The elongation increased more than five times from 3.1% to 15.6%. This finding differs from the extruded state of AZ91 alloy where improvement of mechanical properties is rather insignificant and ambiguous [10]. Such difference is caused by the texture influence. After extrusion, the texture is distinctive and its weakening occurred during ECAP process [5, 6, 10]. Contrary to this, improvement of mechanical properties owing to ECAP of the as-cast alloy was expected [3, 5, 11, 12] due to weak specific grain orientation in this state, significant grain refinement via dynamic recrystallization and much higher dislocation density gained by the used SPD method (i.e. strengthening by dislocation (sub)grain boundaries).

The value measured average of microhardness, 89 HV 0.05, confirmed the findings about partial contribution of the individual areas with different grain sizes to the overall hardness. The average hardness determined by area weighted average from the local microhardness measurements with consideration of both areas ratio (CG area -20%, FG area - 80%, total calculated microhardness -87.6 HV) is in good agreement with the obtained overall hardness. It is worth noting that particles distribution of $Mg_{17}Al_{12}$ also contributes to differences in microhardness between fine and coarse grained areas. This partial contribution of different areas to overall mechanical properties is reached for tensile properties, when the fine grains contribute to the overall strength whereas the coarse grains contribute rather to overall ductility expressed by the elongation during tensile test [13].

5. Conclusions

Based on the results, it is obvious that the ECAP process improves the mechanical properties of the investigated AZ91 alloy. Proof stress $\sigma_{0,2}$ and ultimate tensile strength were improved almost twice by ECAP and the elongation was improved five times comparing to the as-cast state.

The microhardness of as-cast state was of 64 HV 0.05. The microhardness of alloy in ECAPed state for coarse grain areas was of 74 HV 0.025 and 91 HV 0.025 for fine grain areas. These values combined with the area fraction of both types of microstructure correspond well with the overall microhardness of 89 HV 0.05. The ECAP process influenced also the fracture behaviour. Grain refinement and introduced internal strain caused changes in micromechanism of fracture. The amount of cleavage facets on the fracture surfaces was reduced on the behalf of ductile dimples. However, the character and the size of the dimples is similar for both states (as-cast and ECAPed).

Acknowledgements

This has work been supported by the European Regional Development Fund in the framework of the research project NETME Centre under the Operational Program Research and Development for Innovation No. CZ.1.5/2.1.00/01.002, and within the project NETME plus (Lo1202), project of Ministry of Education, Youth and Sports under the "national sustainability programme".

References

- B.L. Mordike, T. Ebert: Mater. Sci. Eng. A302 (2001) 37–45.
- [2] A. Azushima, R. Kopp, A. Korhonen, D.Y. Yang,
 F. Micari, G.D. Lahoti, P. Groche,
 J. Yanagimoto, N. Tsuji, A. Rosochowski,
 A. Yanagina: CIRP Annals Manufacturing Technology 57(2) (2008) 716–735.
- [3] A. Yamashita, Z. Horita, T.G. Langdon: Mater.

Sci. Eng. A300 (2001) 142-147.

- [4] R.B. Figueiredo, T.G. Langdon: J. Mater. Sci. 45(17) (2010) 4827–4836.
- [5] Z. Zúberová, L. Kunz, T.T. Lamark, Y. Estrin, M. Janeček: Metall. Mater. Trans. A38(9) (2007) 1934–1940.
- [6] S.M. Masoudpanah, R. Mahmudi: Mater. Des. 31(7) (2010) 3512–3517.
- [7] S. Fintová, L. Kunz: J. Mech. Behav. Biomed. Mater. 42 (2015) 219–228.
- [8] S.H. Kang, Y.S. Lee, J.H. Lee: J. Mater. Process. Technol. 201(1-3) (2008) 436–440.
- [9] S. Fintová, L. Pantělejev, L. Kunz: Mater. Sci.

Forum 782 (2014) 384–389.

- [10] R. Štěpánek, L. Pantělejev, O. Man: In METAL 2015, Brno, 3rd-5th Jun 2015 [online]. 2015 [cit. 28th October 2015] Available from: http://www.metal2015.com/files/proceedings/21 /papers/3935.pdf.
- [11] M. Janeček, M. Popov, M.G. Krieger, R.J. Hellmig, Y. Estrin: Mater. Sci. Eng. A462 (2007) 116–120.
- [12]Z. Zhao, Q. Chen, H. Chao, C. Hu, S. Huang: Mater. Des. 32(2) (2011) 575–583.
- [13]B. Chen, D.L. Lin, L. Jin, X.Q. Zeng, C. Lu: Mater. Sci. Eng. A483–484 (2008) 113–116.



This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivs 3.0 Unported License. To view a copy of this license, visit http://creativecommons.org/licenses/by-nc-nd/3.0/ or send a letter to Creative Commons, 444 Castro Street, Suite 900, Mountain View, California, 94041, USA.

166 J. Komačka, Z. Florková, J. Mužík: Differences in the surface texture of aggregate particles determined by 3D model derived from optic microscope measurements

DIFFERENCES IN THE SURFACE TEXTURE OF AGGREGATE PARTICLES DETERMINED BY 3D MODEL DERIVED FROM OPTIC MICROSCOPE MEASUREMENTS

Jozef Komačka^{1,*}, Zuzana Florková¹, Juraj Mužík²

- ¹ Department of Highway Engineering, Faculty of Civil Engineering, University of Žilina, Univerzitná 8215/1, 010 26 Žilina, Slovak Republic
- ² Department of Geotechnics, Faculty of Civil Engineering, University of Žilina, Univerzitná 8215/1, 010 26 Žilina, Slovak Republic

*corresponding author: tel. +421415135949, e-mail: komacka@fstav.utc.sk

Resume

The surface texture of aggregate particles was investigated based on the 3D model of surface generated from the measurements by optic microscope. New software MicroSYS was developed to determine the wrapping plane of 3D model of aggregate using the function called "Thin plate spline". New parameter for evaluation of surface texture of aggregate particle was proposed as the volumetric difference between two planes (wrapping plane and aggregate surface).

Applicability of this parameter was tested on two aggregate fractions (4/8 and 8/11) coming form 11 quarries in Slovakia. The tested aggregates differed from petrography point of view and ranged from soft to hard. The difference among the quarries and also between fractions of aggregate was found out. The better surface texture was observed for the finer fraction of aggregate. Simultaneously, the better results were determined in the case of aggregate produced from the igneous intrusive or extrusive rocks comparing to the sedimentary carbonate rocks aggregate.

Article info

Article history: Received 13 September 2015 Accepted 23 December 2015 Online 31 December 2015

Keywords: Surface; Texture; Aggregate; 3D model; Microscope.

Available online: http://fstroj.uniza.sk/journal-mi/PDF/2015/19-2015.pdfISSN 1335-0803 (print version)ISSN 1338-6174 (online version)

1. Introduction

The texture of pavement surface relates to a friction on a tyre/road interface and subsequently influences traffic safety. The positive or negative influence of surface texture ranges (micro-, macro- and megatexture) in relation to a tyre/road friction is defined in [1]. The significance of the individual ranges of texture on the total level of the friction is described in [2]. It is known the micro- and macrotexture are very important from traffic safety point of view. Taking into account their wavelength [1], the size, shape and surface texture of aggregate particles forming the aggregate gradations of various of asphalts predetermine types the level

of micro- and macrotexture of pavement surface. Therefore, the methods detecting mentioned parameters (especially the surface texture) are necessary.

The method using the angularity of aggregate particle is described in [3] and [4]. It has been concluded the more shaped surface of the aggregate, firmer and also sharper material of surface means that better and more lasting friction can be expected. The possibility to measure the surface texture in the range of micro- and macrotexture on pavements in service was tested in [5]. The contactless method using laser scanning with the sensors frequency of 62.5 kHz and 1 kHz was used. Problems with optical illusion

were identified and the authors concluded the more suitable approach is to use the contactless digital image analysis methods (DIAM). Various approaches scanner, (laser photogrammetry, photos and microscope) can be used to obtain a digital image of surface texture that is obviously transferred into 3D. The use of a laser scanner for 3D image of aggregate particle and shape properties evaluation is presented in [6]. High-resolution photos of surface were used in [7] for determination of 3D model of pavement surface. The computer program developed to evaluate the texture showed good correlation with the laser scanning and the pendulum test results. A video or camera is used to take photos for 3D image generation the complex systems developed in for evaluation of aggregate surface texture [8 -12]. Although 3D surface models of aggregate particle are available the texture evaluation is mostly based on the shape of aggregate particle [4, 13 - 15] or an analysis of 2D profiles [11, 16 - 19].

Despite the fact that 2D profiles can be used to determine 3D (volumetric) parameters, the use of 3D model of surface texture increases representativeness of results larger reference area reduce and a an uncertainty due to the random choice of the profile. Information regarding the evaluation of aggregate surface texture using volumetric characteristic derived from the 3D model of aggregate surface could be found rarely. The example is the evaluation presented in [20] where the 3D model of aggregate particle resulting from the photogrammetry method was used. Only top parts of aggregate particles were included evaluation of into the the texture. The parameter Vmp (Peak Material Volume) determined profiles was from based the Abbott-Firestone curve. Lack on of knowledge has been impulse an for the research focused on this topic with the aim to find a way how to distinguish

the quality levels of surface texture of aggregate particle using the volumetric parameter determined from the 3D model of surface texture.

167

2. Experimental

2.1 Generation of 3D model of surface texture

The optic microscope NIKON AZ 100 Elements and the software NIS used Documentation were record to the surface of aggregate. The measurements performed with were the optical magnification of 2 and the lens magnification of 0.5 that resulted in the total magnification of 12.5. The aggregate particle was placed on the pad of the microscope illuminated by the light of microscope and centred in the visual field of microscope. The magnification and quantity of illumination was set up (balance of white colour was always used in this phase). Focusing on the top and bottom of aggregate particle (the pad of microscope was moved up and down) determined the scan range was divided into 400 steps. Thereafter, scanning was run and the recorded data was used to generate the 3D model of aggregate that was exported into the wrml and jpg formats.

2.2 Determination of volumetric parameter for evaluation of texture

The surface texture of aggregate particles was assessed using the software MicroSYS that has been developed especially for this purpose. The basic input is a 3D model of aggregate particle as the output of measurement by microscope (Fig. 1, top). The surface of aggregate is expressed in the analytical form and for the evaluation purpose is replaced by a wrapping plane that is a convex approximation of the analytical function determining the surface of aggregate. It is an iterative process where only the concave parts of analytical function are chosen and replaced. The radial

base function called "Thin plate spline (TPS)" used in the approximation process. is Thereafter, the area of interest of assessed aggregate particle is divided into local areas with the concave course of analytical function. The values of wrapping function are calculated for all local concave areas. Finally, these are connected into the final approximation function that covers the assessed surface of aggregate. The stiffness of TPS function is optimized using null difference between the values of analytical function of aggregate surface and wrapping function at the borderlines of the local concave areas. The result of this process is a wrapping plane of aggregate surface chosen for evaluation (Fig. 1, bottom).



Fig. 1. 3D model of aggregate particle in the software MicroSYS without (top) and with (bottom) the wrap plane.

The volumetric difference between two planes (wrapping plane and aggregate surface) can be used as a parameter for evaluation of surface texture of aggregate particles. The value of difference in volume relates to number of local concave areas and difference in height inside these ones. Lower values express a less propitious texture (small difference between the minimal value at a local area and the value at the borderline of a local area) and vice versa.

2.3 Tested aggregate

The procedures described above were used to difference determine the in volumes for the aggregate coming from 11 quarries in Slovakia. The chosen quarries produce the highest quality aggregate that fulfil all quality criteria required from road construction point of view. The aggregates differed from petrography point of view and ranged from soft (dolomite) to hard (andesite). The overview is done in Table 1. Two fractions (sizes) of aggregate particles, 4/8 and 8/11, taken from each quarry were used in investigation. These fractions are the most important to assure the sufficient level of microtexture of pavement wearing course. It was supposed the different nature of aggregate in the individual quarries (and various portion of hard and soft minerals in these rocks) in combination with equipment used for aggregate production could influence number, shape and evenness of fracture Rightness of this assumption can planes. be observed on the pictures in Table 1. It is evident that the aggregate particles from some localities have their surface rougher when compared to others (e.g. No. 6 and/or 7 compared to No. 8). Likewise, the borderlines of aggregate particle volume are slightly different. Some of them are relatively even others are more bumpy. These entire predispositions resulting from different petrology and production should lead to difference in micro- and macrotexture and the goal of the investigation was whether the volumetric parameter determined according the method described above could be used to reveal it.

		Table 1
	Aggregate used	in testing.
Quarry	Petrographic	Aggregate
No.	type	picture
1	granodiorite	
2	melaphyre	
3	paleobasalt	
4	siliceous limestone	
5	andesite	
6	andesite	
7	paleobasalt	
8	dolomite	
9	andesite	
10	andesite	
11	dolomitic limestone	

12 States

2.4 Results and discussion

The volumetric difference between two planes was determined for each tested aggregate particle using the procedure mentioned above. Five aggregate particles were used in each group representing by a quarry and of aggregate. The determined fraction differences in volumes were transformed to differences in percentage of volume. The approximately same volume of investigated aggregate was chosen for all aggregate particles to have comparable results. It was ensured by position of the base plane (Fig. 1, bottom) that can be positioned at any level in the vertical direction.

The average values were calculated and used for next comparison. As it can be seen in Fig. 2, the volume differences of aggregate are not the same and therefore a difference in surface texture can be supposed. Two basic distinctions be observed. There can is a difference among the quarries and also between fractions of aggregate. The former was expected, reasons mentioned the are in the Chapter 2.3. However, when two fractions are compared, a production equipment should not play any role (obviously the same is used for production both fractions) and a texture of surface should only depend on properties of rock. Nevertheless, it is evident in Fig. 2 the higher value of difference in volume were determined in the case of finer fraction (4/8). According to the used evaluation method, the higher difference in volume means the surface is rougher and aggregate particle has the better texture (microand macro). A reason for it can be that more fracture planes are generated during crushing the aggregate to finer fractions, they overlap into each other and increase an angularity of aggregate. Finally, it could influence evaluation of surface texture based on the volumetric parameter (i.e. difference in volume).

To asses importance of a production equipment comparing to aggregate nature the variability of volume difference was checked using the minimum, maximum and average values (Fig. 3). Based on this it seems the nature of aggregate is more important since the differences in volumes among quarries are a little bit higher than it is in the case of fractions.

Therefore, ranking from the best to the worst was made using the highest value of difference in volume (higher value means better surface texture) for a quarry (regardless fraction of aggregate) to look into the type of rock mined in the individual carries (Fig. 4). It is evident the best values (i.e. the highest values of difference in volume) have the aggregate produced from the igneous intrusive or extrusive rocks (basalt, andesite, melaphyre, diorite). The differences in this group for the same rock (e.g. the quarries No. 5, 6, 9 and 10) relate probably to geologic era and the same type of rock from petrography point various mineralogical of view can have compositions that could influence the brittleness of rock and subsequently the surface texture of aggregate. The worst results (i.e. the lowest values of difference in volume) were found out for the sedimentary carbonate rocks where the silicious and dolomitic limestone were comparable and the dolomite form the quarry No. 8 had absolutely the lowest value of volume difference.



Fig. 2. Volume differences among fractions and quarries.



Fig. 3. Range of differences between the volumes of fractions and quarries.



Fig. 4. Ranking in relation to petrography of aggregate.

3. Conclusions

The surface texture of aggregate particles was investigated based on the 3D model of surface generated from the measurements by optic microscope. New software MicroSYS was developed to determine the wrapping plane of 3D model of aggregate using the function called "Thin plate spline". New parameter for evaluation of surface texture of aggregate particle was proposed as the difference in volume between two planes (wrapping plane and aggregate surface).

Applicability of this parameter was tested on two aggregate fractions (4/8 and 8/11)coming form 11 quarries in Slovakia. The tested aggregates differed from petrography point of view and ranged from soft to hard. The difference among the quarries and also between fractions of aggregate was found out. The better surface texture (i.e. the higher values of difference in volume) was observed for the finer fraction of aggregate. Simultaneously, the better results from surface texture point of view (higher values of difference in volume) were determined in the case of aggregate produced from the igneous intrusive or extrusive rocks comparing to the sedimentary carbonate rocks aggregate.

Based on the results obtained so far, it seems the new approach based on the volumetric difference between two planes of 3D model of aggregate has a potential to distinguish a surface texture of aggregate. Moreover, some relation between this parameter and aggregate nature was observed. To confirm these findings next test have to be carried out to extend the current database of results.

Acknowledgements

and air pollution

The paper is a result of research supported by European regional development fund and Slovak state budget by the project "Research centre of University of Zilina", ITMS 26220220183 and the research VEGA 1/0804/12 Influence of composition of asphalt on characteristics



project

material

of pavement surface texture, noise emission

References

- CEN, EN ISO 13473-5. Characterization of pavement texture by use of surface profiles. Part 5: Determination of megatexture. (2009) Brussels: CEN. CEN, EN ISO 13473-5. Characterization of pavement texture by use of surface profiles. Part 5: Determination of megatexture. (2009) Brussels: CEN.
- [2] M. Kováč, E. Remišová, M. Decký, D. Ďurčanská, J. Čelko: Diagnostika parametrov prevádzkovej spôsobilosti vozoviek (*Diagnostics of* serviceability parameters of pavements), EDIS, Žilina 2012 (in Slovak).
- [3] Y. Kim, L. Souza: Effects of Aggregate Angularity on Mix Design Characteristics and Pavement Performance. [Final report]. Nebraska Transportation Center, Report #MPM-10, 2009.
- [4] E. Masad, T. Al-Rousan, J. Button, D. Little, E. Tutumluer: Test Methods for Characterizing Aggregate Shape, Texture, and Angularity. [Final report – appendixes]. National Cooperative Highway Research Program, Appendixes to NCHRP Report 555, 2005.
- [5] S. Li, S. Noureldin, K. Zhu: Safety Enhancement of the INDOT Network Pavement Friction Testing Program: Macrotexture and Microtexture Testing Using Laser Sensors. [Final report]. Purdue University, Report No. FHWA/IN/JTRP-2010/25, 2010.
- [6] J.K. Anochie-Boateng, J.J. Komba, G.M. Mvelase: Construction and Building Materials 43 (2013) 389–398.
- [7] A. B. Slimane, M. Khoudeir, J. Brochard, M-T. Do: Wear 264(5-6) (2008) 464-468.
- [8] N. H. Maerz, W. Zhou: Flat and Elongated: Advances Using Digital Image Analysis. Proceedings of the 9th Annual Symposium of the International Centre for Aggregates Research (ICAR), Austin, TX, 2001.
- [9] E. Tutumluer, C. Rao, J. Stefanski: Video Image Analysis of Aggregates. [Final Project Report], Civil Engineering Studies UILU-ENG- 2000-2015, University of Illinois Urbana-Champaign, Urbana, IL, Report No. FHWA-IL-UI-278, 2000.
- [10] C. Rao, 2001. Development of 3-D Image

Analysis Techniques to Determine Shape and Size Properties of Coarse Aggregate. [Ph.D. Dissertation], Department of Civil Engineering, University of Illinois at Urbana-Champaign, Urbana, IL, 2001.

- [11] E. Masad: The Development of a Computer Controlled Image Analysis System for Measuring Aggregate Shape Properties. National Cooperative Highway Research Program NCHRP-IDEA Project 77. [Final report]. Transportation Research Board, National Research Council, Washington, D.C, 2003.
- [12] H. Kim, C. Haas, A. Rauch, C. Browne, 2001. A Prototype Laser Scanner for Characterizing Size and Shape Properties in Aggregates. Proceedings of the 9th Annual Symposium of the International Centre for Aggregate Research (ICAR), Austin, TX, 2001.
- [13] S. Tafesse, S. Wenjuan, F. Joanne, L. Wang: A new image analysis technique to quantify particle angularity.http://kth.diva-portal.org/smash /get/ diva2:499535/FULLTEXT01, available online on 29.12.2015.
- [14] T. Al-Rousant, E. Masad, E. Tutumluer, T. Pan: Construction and Building Materials 21 (2007) 978-990.
- [15] Z. Florková: Mikrotextúra hrubého kameniva pre asfaltové zmesi. (*Microtexture of coarse* aggregate for asphalts). [Diploma thesis]. University of Žilina, Žilina 2012 (in Slovak)
- [16] C. Rao, E. Tutumluer, I. T. Kim: Transport. Res. Record 1787 (2002) 117-124..
- [17] E. Masad, D. Olcott, T. White, L. Tashman, L, 2001. Transport. Res. Record 1757 (2001) 148–156.
- [18] T. Fletcher, C. Chandan, E. Masad, K. Sivakumar, J. Testing and Evaluation 30(6) (2002) 524-531.
- [19] Z. Florková, J. Komačka: Procedia Engineering 111 (2015) 209-214.
- [20] G. McQuaid. P. Millar, D. Woodward, S. Friel: Use of close range photogrammetry to assess the micro-texture of asphalt surfacing aggregate. http://www.ijpavement.com/wp-content/uploads/ 2013/12/2013-IJPC-219-2.pdf, available online on 29.12.2015.



This work is licensed under the Creative Commons Attribution-NonCommercial-NoDerivs 3.0 Unported License. To view a copy of this license, visit http://creativecommons.org/licenses/by-nc-nd/3.0/ or send a letter to Creative Commons, 444 Castro Street, Suite 900, Mountain View, California, 94041, USA.

P. Singh, A. Handa: Mechanical Properties of Stellite-6 coated AISI 316L Stainless Steel

173

MECHANICAL PROPERTIES OF STELLITE-6 COATED AISI 316L STAINLESS STEEL

Pushpinderjit Singh^{1*}, Amit Handa¹

¹Department of Mechanical Engineering, RIMT- Institute of Engineering & Technology, Mandi Gobindgarh-147301, Punjab, India.

*corresponding author: er.pushpinderjit@gmail.com

Resume

Present paper describes the mechanical properties of Stellite-6 coated AISI 316L stainless steel. Specimens were coated using Detonation Gun thermal spray process, with different coating thicknesses of Stellite-6 ranging from 50 μ m to 150 μ m. Afterwards their properties like tensile strength, impact strength and micro hardness were evaluated on the basis of the results obtained from the experimentation. For comparison of substrate and coated material, graphs were plotted. The coated specimens exhibited superior impact strength and microhardness than that of the bare specimens, whereas the tensile strength of coated specimens decreased marginally with the increase in coating thickness.

Article info

Article history: Received 31October 2015 Accepted 12 January 2016 Online 31 January 2016

Keywords: Thermal spray coating; Tensile strength; Charpy impact test; Stellite-6 alloy; Microhardness

ISSN 1335-0803 (print version) ISSN 1338-6174 (online version)

1. Introduction

In a wide variety of applications, mechanical components have to operate under severe conditions, such as high load, speed and hostile chemical or temperature environment. Thus, their surface modification is necessary in order to protect them against various types of degradation. Thermal spraying has emerged an important as tool of increasingly sophisticated surface engineering technology. The different properties of the coating provide corrosion resistance, wear resistance, better thermal or electrical conductivity etc. [1]. AISI 316L austenitic stainless steel is widely used in corrosive environments as it has high corrosion resistance. However, its wear, cavitation and erosion resistances are poor due to low hardness (220 HV), which restricts its use in many industrial applications as reported by Viswanathan et al. [2]. Austenitic stainless steel exhibits a sticking behaviour during wear test, which

is a distinctive feature of ductile materials that are capable of absorbing much larger quantities of energy before failure as found by Dalmau et al. [3].

Coating a material with another material is done to obtain a combination of properties that combine the properties of the base material with other necessary properties that give corrosion resistance, wear resistance, better thermal or electrical conductivity as reported by Molleda et al. [4].

Stellite-6 is the most widely used cobalt based alloy exhibiting excellent resistance to many forms of mechanical & chemical degradation. Its applications include valve seats & gates, pump shafts, bearings and erosion shields. The exceptional wear resistance of Stellite-6 is mainly due to the unique and inherent characteristics of the hard carbide phase dispersed in Co-Cr alloy matrix as found by Prasad Rao et al. [5].

This paper deals with influence of Stellite-6 coating on AISI 316L stainless steel

and on mechanical properties viz. tensile strength, impact strength and microhardness.

2. Experimental Material & Procedure

Stellite-6 alloy in powder form was used as coating material, the nominal composition of which is shown in Table 1.

The substrate material used was AISI 316L stainless steel. AISI 316 L Stainless Steel is extra low carbon version of 316 steel alloys. The low carbon content in AISI 316 L Stainless Steel prevents the formation of carbide on the surface when material comes in contact of heat. This can be used for bearings which operate in corrosive environments such as running inside liquids. But this steel has slightly lower mechanical properties. This steel fails mainly due to wear and elevated temperatures.

The chemical composition of substrate material is given in Table 2.

Before coating, the material was machined and different specimens for tensile, impact and micro hardness tests were prepared.

2.1 Fabrication of microhardness test specimens

For microhardness test, specimens of Dia. 20 mm x 20 mm length were prepared by cylindrical turning on lathe machine. Both flat sides of the specimens were ground on a surface grinder. Fig. 1 depicts the specimens for micro hardness test.



Fig. 1. Specimens for Micro hardness test.

2.2 Fabrication of Charpy impact test specimens

The specimens for Charpy impact test were prepared on a milling machine and dimensions kept according to specifications of standard ASTM A370. Firstly the samples were roughly cut and then were ground on a surface grinder to make the sides truly perpendicular. Three specimens for each coating with dimensions of 10x10x55 mm were prepared so as to maintain repeatability of test results. The notch with depth of 2 mm was made in the centre of specimens using a wire cut machine. The included notch angle was kept 45°. Fig. 2 depicts the impact test specimens.



Fig. 2. Impact Test Specimens.

2.3Fabrication of tensile test specimens

The raw material was machined on lathe machine and tensile test specimens were prepared according to standard ASTM E8. The gauge length was ground to 50 μ m. The gauge length diameter was kept 12.5 mm for bare specimen. For specimens which were to be coated, the gauge length diameters were kept 12.4 mm, 12.3 mm and 12.2 mm for coating thickness 50 μ m, 100 μ m and 150 μ m respectively. Three specimens for each coating were prepared to maintain repeatability of results. Fig. 3 shows the machined specimens (prior to coating) for tensile test.



Fig. 3. Tensile Test Specimens.

									1 ubie 1
			The che	mical comp	position of S	Stellite-6.			
Element	Cr	W	С	Ni	Fe	Si	Mn	Mo	Со
wt. %	28.60	4.90	1.30	0.65	0.79	1.25	1.13	< 0.10	Bal.
									Table 2
			Chemi	cal compos	sition of AIS	SI 316 L			
Element	С	Si	Mn	Р	S	Ni	Cr	Mo	Fe
wt. %	0.02	0.40	1.32	0.026	0.03	10.27	16.57	2.01	Bal.

Table 3

Technical Specification of Awaaz Detonation Gun.						
Working Gases	Oxygen, Acetylene, Nitrogen and Air					
Consumption of Powder per Shot	0.02-0.05g/ shot					
Water Consumption Rate	15-25 litres/minute					
Firing Rate	1-10 Hz					
Coating Thickness per Shot	5-25 μm					
System Control	Manual/ Semi auto					
Dimensions (L X B x H) mm	1200 x 500 x 1500					
Sound Pressure Level	150 dB					

Table 4

Process parameters	for	Stellite-6	coating	by D-	gun	thermal	spray	process.
				/	()		··· ··· ·	

Parameter	Value	
Oxygen Flow Rate	3120 SLPH (Standard Litres Per Hour)	
Pressure	0.2 MPa	
Acetylene Flow Rate	2400 SLPH (Standard Litres Per Hour)	
Pressure	0.14 MPa	
Nitrogen Flow Rate	1040 SLPH (Standard Litres Per Hour)	
Pressure	0.4 MPa	
Spray Angle	90°	
Spray Distance	150 mm	
Power	450 VA	

2.4 Coating of specimens

For coating process the equipment used was "Awaaz Detonation Gun" available at M/s SVX Powder M Surface Engineering Pvt. Ltd., Greater Noida (India).

The technical specifications of the detonation gun are given in Table 3.

The process parameters for thermal spray Stellite-6 coating by detonation gun are given in Table 4. The specimens to be coated were firstly sand blasted using alumina powder (Al_2O_3) to make the surface suitable for coating deposition. Fig. 4 shows different coated specimens.





b) Charpy impact test specimen

2

c) Tensile test specimen. Fig. 4. Different coated Specimens.

Tabla1

2.5 Testing of specimens

Micro Hardness test: The bare and coated specimens were tested on micro Vickers hardness tester available at Research and Development Centre for Bicycles and Sewing Machines, Ludhiana.

Charpy impact test: The bare and coated specimens for Charpy impact test were tested on Charpy impact test machine available at Research and Development Centre for Bicycles and Sewing Machines, Focal Point, Ludhiana. The impact testing was done using ASTM A370 standard. Fig. 5 shows the specimens after Charpy impact test.





a) Bare AISI 316L specimen







c) Specimen with 100µm Stellite 6 coating Fig. 5. Specimens after Charpy impact test.



Fig. 6. Tensile test being performed on UTM. (full colour version available online)

Tensile Test: The bare and coated specimens for tensile test were tested on FIE make Universal Tensile Testing machine (UTE-100) of 100 ton capacity, available at CITCO-IDFC Testing Laboratory, Chandigarh. The tensile testing was done using ASTM E8/E8M standard. The gauge length was kept 60 mm and gauge points were marked by using dot punch. The dumbbell shaped specimens were tightened in the jaws of the UTM as depicted in Fig. 6.

The load was applied gradually and readings were taken. Fig. 7 shows the specimens after tensile test.



c) Specimen with 100µm Stellite 6 coating

Coating showing brittle fracture



d) Specimen with 150µm Stellite 6 coating Fig. 7. Specimens after tensile test.

3. Results and discussion

3.1 Micro hardness test

А drastic difference found was in the microhardness specimens. of the The average hardness of specimen with150 µm thick coating of Stellite-6 at 653 HV_{0.5} was significantly higher than that of the substrate at average value of 249 $HV_{0.5}$. The microhardness profile is shown in Fig. 8.



Microhardness profile of Stellite-6 coated Specimens

ig. 8. Microhardness profile across coated and substrate regior (full colour version available online)

The microhardness of the specimen with 150 μ m Stellite-6 coating increased by more than 200 % as compared to the substrate region of AISI 316L. It was inferred that the increase is not only due to presence of chromium carbide particles in micron, but due to formation of tiny particles within the coating as found by Apay and Gulenc [6].

3.2 Charpy impact test

The energy consumed by the bare specimens was lower than that of the coated specimens. The bare specimens consumed 156 kJ of energy whereas the specimens with 150 µm Stellite 6 coating absorbed 216 kJ of energy. Therefore distinct behaviour was observed. The bare specimens showed less impact strength and the coated specimens showed better impact strength. The crack was made to propagate first through the Stellite-6 coated section followed by substrate. Similar investigations have been carried out by Ganesh et al. [7]. The impact strength went on increasing with the increase in coating thickness. Fig. 9 shows the variation in impact strength with respect to coating thickness.

3.3 Tensile test

The results of tensile test revealed that the bare AISI 316L specimens showed ductile failure. Necking took place in the gauge section and the specimen failed at a high value of UTS. However the specimens with Stellite-6 coating showed different behaviour. The coating on the specimens showed a brittle fracture and coating got detached from substrate thereby exposing the bare stainless steel beneath. This was followed by ductile failure of the substrate. The tensile strength went on decreasing with increase in coating thickness. The specimen of bare AISI 316L had the maximum tensile strength and the one with 150 µm had the lowest tensile strength. This may be due to the effect of heat during the surface treatment which may have resulted in microstructural changes. But the decrease is only marginal. The specimens with coating thickness of 150 µm had an average tensile strength of 0.681 kN.mm⁻² whereas the bare specimens of AISI had an average tensile strength of 0.726 kN.mm⁻². Therefore the decrease in tensile strength is only about 7 % which is negligible. Fig. 10 shows the tensile behaviour of bare and coated specimens.



Fig. 9. Graph showing relationship between impact strength and coating thickness. (full colour version available online)



Tensile Strength vs Coating Thickness

Fig. 10. Graph showing relationship between tensile strength and coating thickness. (full colour version available online)

4. Conclusions

In the light of the results obtained during the course of present investigation, it is inferred that:

• The Stellite-6 coating, on AISI 316 L, done by D-gun process resulted in great increase in micro hardness which can be attributed to Co-Cr matrix formed in the coated section.

• The impact strength also improved as the thickness of Stellite-6 coating went on increasing. This is due to crack propagation through the coating section first and then going through the stainless steel substrate region.

• The increase in thickness of Stellite-6 coating resulted in marginal decrease in tensile strength. Detachment of the coating material occurred during tensile test which exposed the ductile substrate material. Therefore application of an interlayer or bond coat was suggested.

•The decrease in tensile strength which is only about 7% can be neglected in the comparison to drastic increments in microhardness and impact strength which increased about 200% and 40% respectively.

References

- B.S. Sidhu, D. Puri, S. Prakash. J. Mater. Process. Technol. 159(3) (2005) 347-355.
- [2] A. Viswanathan, D. Sastikumar, H. Kumar, A.K. Nath. Surf. Coat. Technol. 203(12) (2009) 1618-1623.
- [3] A.Dalmau, W.Rmili, D. Joly, C. Richard, A. Igual-Munoz. Tribology Letters 56(3) (2014) 517-529.
- [4] F. Molleda, J. Mora, F.J. Molleda, E. Mora, E. Carrillo, B.G. Mellor. Mater. Char. 57(4-5) (2006) 227-231.
- [5] K. Prasad Rao, R. Damodaram, H. Khalid Rafi, G.D. Janaki Ram, G. Madhusudhan Reddy, R. Nagalakshmi. Mater. Char. 70 (2012) 111-116.
- [6] S. Apay, B. Gulenc. 2014, Mater. Des. 55 (2014) 1-8.
- [7] P. Ganesh, A. Moitra, P. Tiwari,

S. Sathyanarayanan, H. Kumar, S. Rai, R Kaul, C.P. Paul,R.C. Prasad, L.M. Kukreja. Mater. Sci. Eng. A 527(16-17) (2010) 3748-3756.

- [8] A.S.C.M. D'Oliveira, P.S.C.P. Da Silva, R.M.C. Vilar. Surf. Coat. Technol. 153(2-3) (2002) 203-209.
- [9] Y. Zou, Ch.-Y. Zhan, B. Yang, J.-Ch. Wu. J. Nucl. Mater. 436(1-3) (2013) 56-60.
- [10] M. Taguchi, H. Sumitomo, R. Ishibashi, Y. Aono. Mater. Trans. 49(6) (2008) 1303-1310.
- [11] M.G. Hebsur, J.J. Moore. Eng. Fract. Mech. 22(1) (1985) 93-100.
- [12]G. Xu, M.Kutsuna, Z. Liu, K. Yamada. Surf. Coat. Technol. 201(3-4) (2006) 1138-1144.
- [13] ASTM 370- 07a "Standard Test Methods and Definitions for Mechanical Testing of Steel Products"
- [14] ASTM E8/ E8M- 09, "Standard Test Methods for Tension Testing of Metallic Materials."