



Nanoindentation Studies of $\text{Al}_2\text{O}_3\text{-TiO}_2$ Coating with NiAl Interlayer Deposited on AlSi7Mg Alloy Casting

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Abstract

The paper presents the results of material properties investigations (nanoindentation test) of an $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating with a NiAl interlayer, deposited by the APS (Atmospheric Plasma Spraying) method on an AlSi7Mg alloy casting. The scope of research included the analysis of the microstructure and chemical composition of the substrate material, powders, and coatings, substrate surface preparation, coating fabrication, and the analysis of nanoindentation test results, determined according to the Oliver-Pharr method. The analysis of the nanoindentation test results indicates significantly higher values of material properties, including hardness and elastic modulus, for the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating and the NiAl interlayer compared to the AlSi7Mg alloy substrate, with a gradual increase in properties observed from the substrate toward the outer ceramic layer. The obtained results confirm the possibility of using the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating as TBC – Thermal Barrier Coatings to improve the operational durability of internal combustion engine components made of Al-Si alloy.

Keywords: AlSi7Mg alloy, APS method, $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating, Nanoindentation test

1. Introduction

Aluminum-silicon alloys, due to their low density, good thermal conductivity, and favorable mechanical and casting properties, are widely used in the construction of internal combustion engine components [1]. The AlSi7Mg alloy belongs to the group of hypoeutectic silumins, characterized by good casting properties and satisfactory strength properties [2,3]. Increasing requirements regarding engine power enhancement and exhaust emission reduction mean that elements of this component operate under very extreme thermal and mechanical conditions. High temperature, variable pressure, and thermal shock caused by the combustion of the fuel-air mixture lead to progressive degradation of the material, creating a risk of permanent damage to the elements

of these components. An additional factor limiting the operational durability of components made of AlSi alloys is their relatively low resistance to plastic deformation [4-6].

One way to minimize damage to engine components caused by thermal load is the use of TBCs (Thermal Barrier Coatings). Among many coating deposition techniques that ensure high adhesion, microstructure homogeneity, and the possibility of precise control over the functional properties of the coating, the APS (Atmospheric Plasma Spraying) method can be distinguished [7,8]. In this method, a ceramic $\text{Al}_2\text{O}_3\text{-TiO}_2$ composite is often used as the outer layer material, in which the addition of TiO_2 improves plasticity and fracture toughness while maintaining high hardness and chemical resistance [9]. Due to the significant difference in thermal expansion coefficients between the ceramic and the metallic substrate, it is necessary to use a metallic bond coat



(interlayer), for example, NiAl. The metallic NiAl interlayer ensures continuity and a good bond with both the ceramic and the substrate material [10].

The aim of the work was to analyze the material properties determined in nanoindentation tests of a ceramic $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating with a NiAl interlayer, deposited by the APS method on the surface of an AlSi7Mg alloy casting. The presented research results are part of a cycle of research and scientific works dedicated to the evaluation of the material properties of TBCs [11, 12], as well as own research dedicated to the possibility of using ceramic coatings to improve the operational durability of internal combustion engine components made of Al-Si alloy [13,14].

2. Research Material and Methodology

The substrate for the coating deposition was an AlSi7Mg alloy casting. The chemical composition analysis was performed using a Q4 Tasman emission spectrometer (Table 1).

Table 1.

Results of the chemical composition analysis of the AlSi7Mg alloy

Si, %	Mg, %	Mn, %	Fe, %	Al, %
6,97	0,26	0,01	0,10	Rest

Microstructure observations were carried out on metallographic specimens, which were ground and polished, and then etched with a 20% NaOH solution. Microstructure analysis was performed using a TESCAN Vega 3 scanning electron microscope. An example view of the AlSi7Mg alloy microstructure is shown in Figure 1.

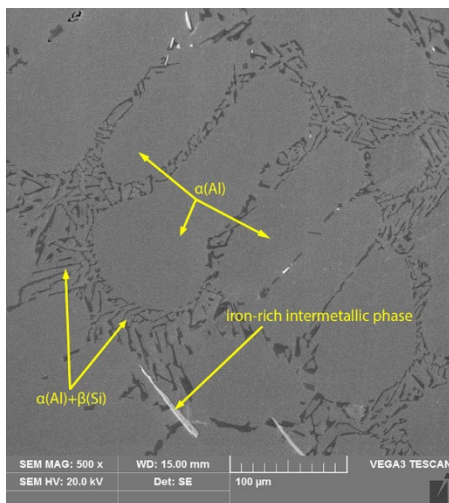


Fig. 1. SEM image of the AlSi7Mg alloy microstructure

The microstructure of the AlSi7Mg alloy contains phases typical for a hypoeutectic silumin, i.e., the proeutectic $\alpha(\text{Al})$ phase, the $\alpha(\text{Al})+\beta(\text{Si})$ eutectic, and intermetallic phases (mainly iron-rich).

Two types of powders were used in the spraying process. The first was used to fabricate the NiAl interlayer, while the second was

used to deposit the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating. The interlayer was made of Metco 404NS powder with a composition containing 80% Ni, and the remainder was aluminum. The particle morphology of the NiAl powder is presented in Figure 2. The powder grains were characterized by a spherical or irregular shape and a particle size distribution in the range of 45–120 μm .

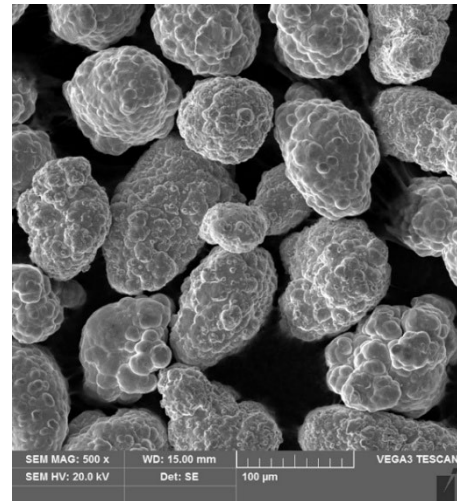


Fig. 2. SEM image of NiAl powder

The powder used to fabricate the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating was characterized by the phase composition: 60% Al_2O_3 and 40% TiO_2 . The particle morphology of the $\text{Al}_2\text{O}_3\text{-TiO}_2$ powder is illustrated in Figure 3. Analysis of the powder particles showed that it consisted of grains with a spherical shape and a particle size distribution of 10–70 μm .

Before the spraying process, the substrate surface was subjected to grit-blasting treatment with corundum grain and degreased in an acetone bath in an ultrasonic cleaner. This treatment aimed to increase the surface roughness to achieve high adhesion of the coating to the substrate material.

The coating deposition process was performed by the APS method. The technological parameters of the spraying process for the NiAl and $\text{Al}_2\text{O}_3\text{-TiO}_2$ coatings are summarized in Table 2.

The nanoindentation test, aiming to determine the material properties values of the substrate and coatings, was performed using a Nanoindentation Tester NHT by CSM Instruments with a Berkovich indenter (B-L 32) and a force of 100 mN. The loading and unloading rate of the indenter was 200 mN/min.

The nanoindentation tests were carried out based on the Oliver-Pharr method [15] and the PN-EN ISO 14577-1:2005 standard.

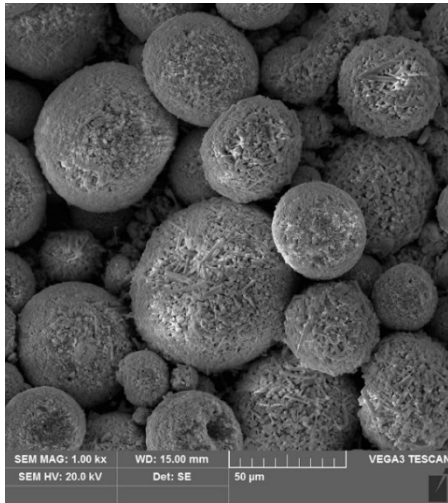


Fig. 3. SEM image of $\text{Al}_2\text{O}_3\text{-TiO}_2$ powder

Table 2.
APS spraying parameters

Process parameters	Powder type	
	$\text{Al}_2\text{O}_3\text{-TiO}_2$	NiAl
Carrier gas (l/min)	4	4
Feeder disk speed (%)	5	5
Current intensity (A)	620	620
Plasma-generating gas Ar (l/min)	38	30
Plasma-generating gas H_2 (l/min)	15	5,2
Air jet – air (bar)	3	3
Spraying distance (mm)	120	100
Torch speed (mm/min)	250	200
Layer thickness (μm)	350-400	120-200
Number of torch passes (-)	20	10

Figures 4 and 5 show a schematic of the indenter loading and unloading process, parameters characterizing the contact geometry between the indenter and the material, and a typical curve of the indenter tip displacement during loading and unloading [15].

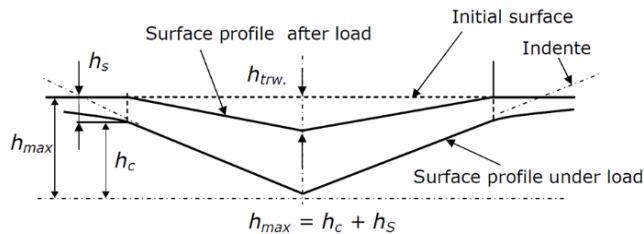


Fig. 4. Schematic of the indenter loading and unloading process and parameters characterizing the contact geometry

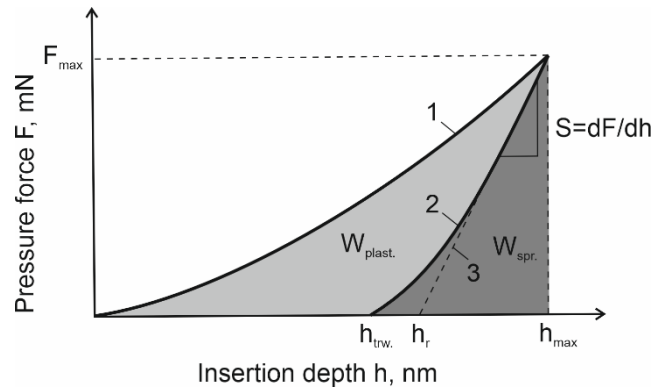


Fig. 5. Typical curve of the indenter tip displacement during loading and unloading

In Figure 5, within the indenter tip displacement curve, section 1 corresponds to the indenter loading, section 2 is the indenter unloading, and section 3 is the tangent to the unloading curve at the maximum load $F_{\max}$. The parameter h_r means the theoretical indentation depth determined assuming a constant value of the elastic unloading stiffness S , as the intersection of the tangent to the unloading curve at the initial unloading stage at point $F_{\max}$ with the abscissa axis. The depth $h_{\max}$ is the maximum indentation depth at the maximum force load $F_{\max}$. The depth h_{trw} defines the indentation depth (permanent deformation) after indenter unloading, while h_c is the depth at which contact occurs between the indenter and the material. The parameter S , the so-called elastic unloading stiffness, is the slope of the upper part of the unloading curve at the initial unloading stage ($S = dF/dh$). The parameters $W_{plast.}$ and $W_{spr.}$ are the plastic deformation work and elastic deformation work, respectively.

The material properties were determined for the $\alpha(\text{Al})$ phase and the $\alpha(\text{Al})+\beta(\text{Si})$ eutectic in the AlSi7Mg alloy (substrate), as well as in the NiAl interlayer and the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating. Three measurements were performed for each microstructure component, and the results were averaged.

3. Results and Analysis

Figure 6 shows a view of the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating with a NiAl interlayer deposited on the AlSi7Mg alloy substrate. Microstructure analysis of the bonding area between the NiAl intermediate layer and the AlSi7Mg alloy substrate showed no material discontinuities. Examination of the boundary between the NiAl coating and the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating also revealed no structural defects, indicating good adhesion of the coating to the interlayer.

Figures 7-10 show example indenter loading and unloading curves during nanoindentation in individual microstructure components of the AlSi7Mg alloy, the NiAl interlayer, and the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating.

The averaged values of the material properties of the AlSi7Mg alloy microstructure components, the NiAl interlayer, and the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating are presented in Table 3.

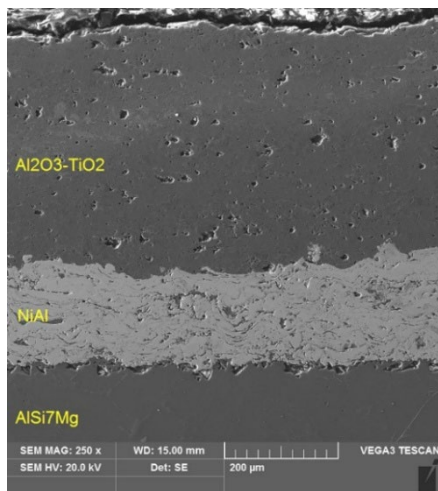


Fig. 6. SEM image of the metallographic specimen of the Al_2O_3 - TiO_2 coating with a NiAl interlayer on the AlSi7Mg alloy casting

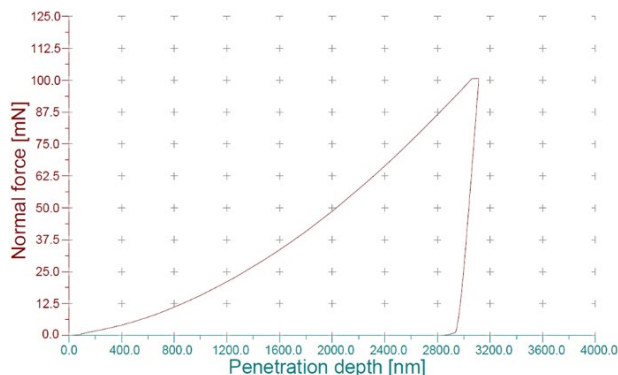


Fig. 7. Indenter loading and unloading curve during nanoindentation of the $\alpha(\text{Al})$ phase in the AlSi7Mg alloy microstructure

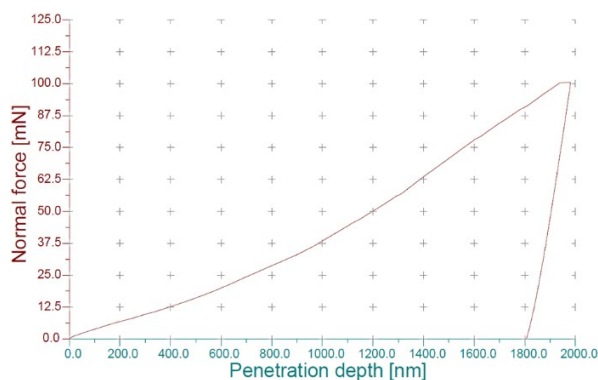


Fig. 8. Indenter loading and unloading curve during nanoindentation of the $\alpha(\text{Al})+\beta(\text{Si})$ eutectic in the AlSi7Mg alloy microstructure

Analysis of Figures 7-10 indicates a different nature of the indenter loading and unloading curves during nanoindentation of the AlSi7Mg alloy, the interlayer, and the Al_2O_3 - TiO_2 coating.

Clear differences are visible especially in the share of plastic work W_{plas} and elastic work W_{elast} in the total mechanical work. The higher share of elastic work W_{spr} in the total mechanical work during indenter indentation into the Al_2O_3 - TiO_2 coating, measured by the value of the η_{IT} parameter, indicates its higher resistance to plastic deformation compared to the substrate material, i.e., the AlSi7Mg alloy. This is also reflected in the values of HV hardness and the HIT hardness determined during indenter indentation. The values of these hardnesses are significantly higher than the hardness of both microstructure components of the AlSi7Mg alloy. In the case of the Al_2O_3 - TiO_2 coating, a clearly higher value of the indentation elastic modulus EIT was also obtained.

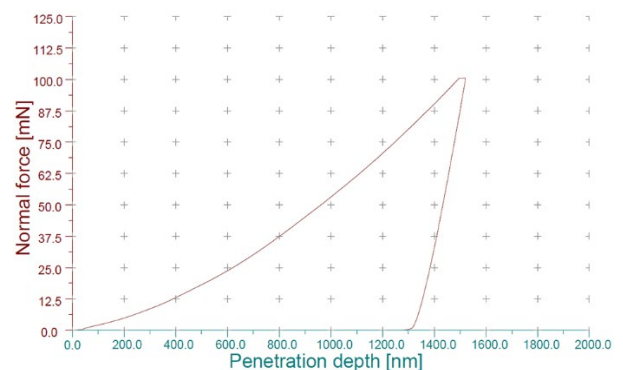


Fig. 9. Indenter loading and unloading curve during nanoindentation of the NiAl interlayer area

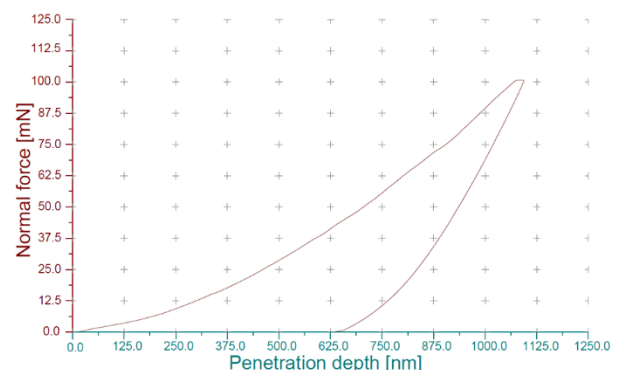


Fig. 10. Indenter loading and unloading curve during nanoindentation of the Al_2O_3 - TiO_2 coating area

The different nature of the indenter loading and unloading curves during indentation of the AlSi7Mg alloy microstructure components and the Al_2O_3 - TiO_2 coating indicates different mechanisms of plastic deformation and material degradation due to the indenter action. Figures 11-14 show example views of the indenter impressions in individual microstructure components of the AlSi7Mg alloy, the NiAl interlayer, and the Al_2O_3 - TiO_2 coating. In the case of the $\alpha(\text{Al})$ phase in the AlSi7Mg alloy, clear pile-ups of material are visible on the edges of the impression, which indicate low resistance to plastic deformation. The indenter impression in the second structural component of the AlSi7Mg alloy is characterized by significantly smaller pile-ups. This is caused by the presence of a hard $\beta(\text{Si})$ phase with low elastic properties, in which cracks may form.

Table 3.

Nanoindentation test results

Parameter	AlSi7Mg substrate		Type of coating	
	$\alpha(\text{Al})$ phase	$\alpha(\text{Al})+\beta(\text{Si})$ eutectic	NiAl	$\text{Al}_2\text{O}_3\text{-TiO}_2$
Fmax – maximum load force (mN)	100,64	100,45	100,71	100,69
hmax – maximum penetration depth (nm)	3128,62	1980,39	1702,86	1093,75
S – elastic unloading stiffness (mN/nm)	0,8507	0,7046	0,6870	0,3623
hc – depth between indenter and material (nm)	3041,87	1866,87	1588,87	886,03
hr – theoretical indentation depth (nm)	3010,33	1837,83	1556,26	815,81
htrw – indentation depth after unloading (nm)	2909,22	1807,75	1512,21	658,29
HV – Vickers hardness (HV)	43,602	116,36	161,12	511,29
HIT – hardness determined during indentation (MPa)	470,81	1256,4	1739,8	5520,8
EIT – indentation elastic modulus (GPa)	47,447	65,305	75,519	70,642
E* – reduced modulus of elasticity (GPa)	52,139	71,764	82,988	77,628
CIT – relative change in indentation depth (%)	1,68	2,16	1,87	1,91
W _{elast.} – elastic deformation work (pJ)	7648,92	7894,53	8414,28	17391,71
W _{plas.} – plastic deformation work (pJ)	112723,08	78181,42	60401,79	25507,81
W _{total.} – total mechanical work (pJ)	120371,99	86075,95	68816,07	42899,52
η_{IT} – elastic component of indentation work (%)	6,35	9,17	12,23	40,54

The SEM image of the indenter impression in the NiAl interlayer is deformed by pile-ups to a much lesser extent compared to the AlSi7Mg alloy substrate material.

The shape of the impression in the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating is irregular. This is related to the build and structure of the coating

deposited using the APS method. In the plasma spraying process, the coating is formed as a result of partial melting or complete melting of the powder particles. Structurally, it is not homogeneous. In its build, one can distinguish the boundaries of grains formed as a result of mechanical bonding of melted or partially melted powder particles accelerated to high velocities in the plasma jet. As a result of the indenter action, these boundaries undergo broadening and discontinuities similar in shape to cracks appear. It can be assumed that the irregular, unidirectional elongated shape of the indenter impression in the $\text{Al}_2\text{O}_3\text{-TiO}_2$ coating is the result of the presence of a bonding boundary of partially melted powder particles in its area.

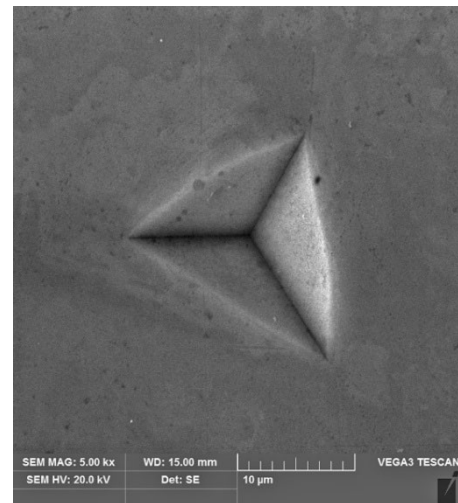


Fig. 11. SEM image of the indenter impression in the $\alpha(\text{Al})$ phase in the AlSi7Mg alloy microstructure

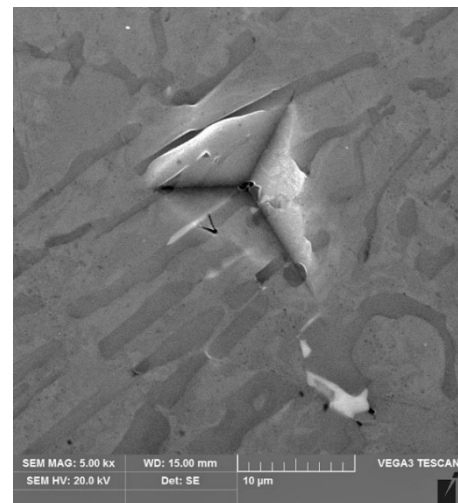


Fig. 12. SEM image of the indenter impression in the $\alpha(\text{Al})+\beta(\text{Si})$ eutectic in the AlSi7Mg alloy microstructure

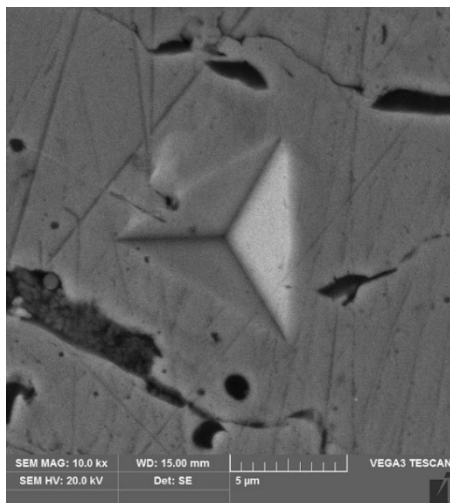


Fig. 13. SEM image of the indenter impression in the NiAl coating

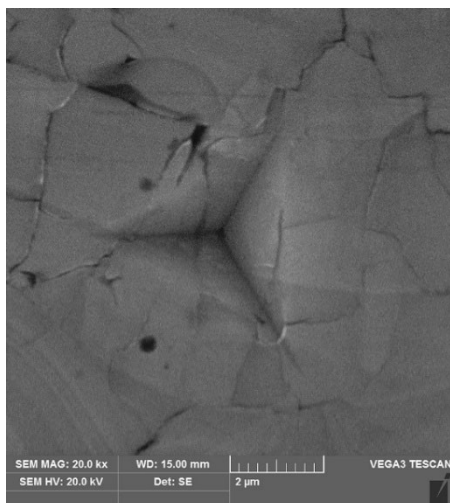


Fig. 14. SEM image of the indenter impression in the Al₂O₃-TiO₂ coating

4. Conclusions

The paper presents the results of investigations into the material properties of an Al₂O₃-TiO₂ coating with a NiAl interlayer deposited on an AlSi7Mg alloy substrate, as determined by nanoindentation testing.

The results of the material property characterization of the Al₂O₃-TiO₂ coating confirm its potential application for improving the service life of internal combustion engine components manufactured from Al-Si alloys. The Al₂O₃-TiO₂ coating exhibits substantially higher material property values than the AlSi7Mg alloy substrate, particularly with respect to its greater resistance to plastic deformation and enhanced elastic properties.

The NiAl interlayer plays a significant role in the deposition process of the Al₂O₃-TiO₂ coating, particularly in terms of

achieving a high-quality bond with the substrate. In the case of plasma spraying, the coating-substrate interface is predominantly adhesive in nature. Achieving a high-quality adhesive bond, without discontinuities at the substrate-coating interface, depends not only on the technological parameters of the plasma spraying process but also on the physicochemical and mechanical properties of both the substrate and the coating materials. When substantial differences exist between these properties, particularly in the coefficients of thermal expansion, there is a high risk of discontinuities, such as cracks, developing at the substrate-coating interface. The application of an interlayer can significantly reduce this risk.

The nanoindentation results presented in this study constitute the first stage of research into the application of Al₂O₃-TiO₂ coatings for improving the service life of internal combustion engine components manufactured from Al-Si alloys. As part of the subsequent stages of the comprehensive research plan addressing this topic, abrasive wear resistance tests will be conducted, with particular emphasis on elucidating the wear mechanisms of both the substrate and the coating. For this purpose, analyses of the surface topography after wear testing, as well as investigations of the microstructure and chemical composition, will also be performed, together with further characterization of the material properties.

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