CSM Instruments
Advanced Mechanical Surface Testing

APPLICATIONS BULLETIN

Characterization of thermal spray coatings by instrumented indentation and scratch testing Part II: Indentation of plasma sprayed coatings

//// Introduction

The first part of this CSM Instruments Application Bulletin was dedicated to the demonstration of the use of instrumented indentation and scratch testing for characterization of HVOF coatings. This second part extends the application of the instrumented indentation method to plasma sprayed ceramics and metals. The paper will focus on advantages of instrumented indentation when applied to plasma sprayed coatings with respect to their heterogeneous microstructure.

//// Instrumented indentation features

The instrumented indentation method reveals much more information about a thermal spray coating than conventional Vickers or Knoop microhardness methods. Though the force range can overlap, the results of instrumented indentation bring more information about mechanical properties of the coatings such as elastic modulus, elastic/plastic work of indentation and creep or cyclic behavior. Due to its force-displacement recording capability, the instrumented indentation method is able to sense when an indentation is performed on a void: the depth suddenly increases without a corresponding increase in force (Fig. 1). Such effects would probably not be noticed in conventional Vickers measurements.

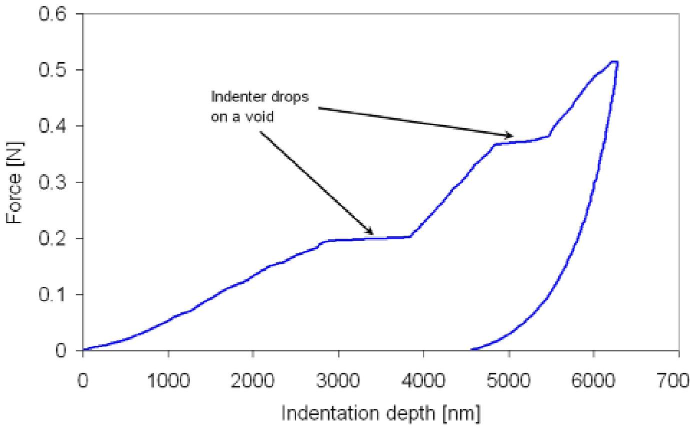


Fig. 1 – Loading-unloading indentation curve presenting two 'indenter drops' due to indentation on voids or other defects..

One of the most significant advantages of instrumented indentation is the possibility of automated measurements due to the hardness and elastic modulus calculation from the loading-unloading curve. The automation can be divided into two types:

> spatial automation (matrix indentation),
> automated indentation load increase (depth profiling).

//// Visual matrix indentation

Visual matrix indentation is a particular case of spatially resolved matrix indentation where indentation sites are located under the microscope and then indented automatically. This type of measurement is especially useful for heterogeneous thermal spray coatings. Most of the results presented in this paper were obtained by the Visual matrix indentation method.

//// Continuous Multi Cycle indentation (CMC)

This efficient cyclic indentation method allows automatic increase of maximum load on each subsequent cycle. Dataset of indentation depth and hardness or elastic modulus is obtained within ten or twenty minutes depending on the number of cycles. This method is particularly effective for studying hardness and elastic modulus of thermal spray coatings at various loads and depths.

//// Indentation parameters

Two CSM Instruments systems were used for characterization of the mechanical properties of the coatings: Nanoindentation Tester (NHT) and Micro Indentation Tester (MHT). The NHT was used in its NHT-TTX standalone version, a full performance nanoindentation system with load range of 0.1 mN to 500 mN equipped with Berkovich indenter, motorized tables and optical microscope. The MHT (load range 0.03 N to 10 N, Vickers indenter) was mounted on a Compact Platform equipped with a confocal microscope and optical microscope. All measurements were done by the CMC method and the indented areas were selected by Visual matrix in most cases. On each sample five to seven measurements of the same type were performed and only average values are presented.

//// Plasma sprayed alumina

The water stabilized plasma (WSP) spraying is a unique technique for high throughput spraying of ceramics and metals. It has been used for many years for spraying of e.g. high quality alumina (Al_2O_3), chromium oxide (Cr_2O_3) and steel coatings. The main advantage of the WSP spraying is high enthalpy of the plasma which allows for spraying of materials with high melting point and high throughput.

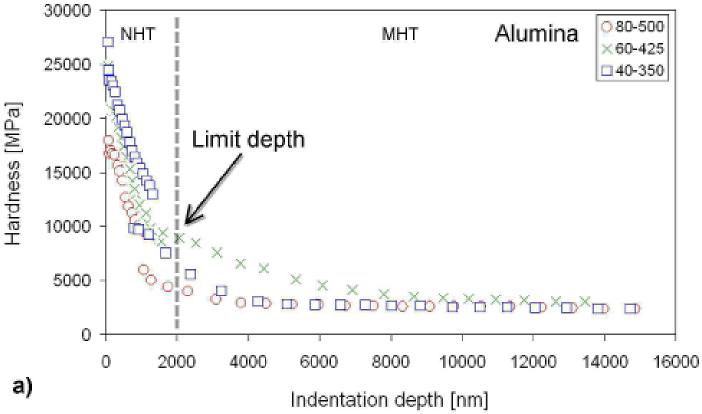
The presented study deals with white alumina sprayed at various feeding and spraying distances. The spraying and feeding distances are essential spraying parameters as they determine the mechanical properties of the material. The coatings were sprayed at the Institute of Plasma Physics (Prague, Czech Republic) on steel substrates. Three combinations of feeding and spraying distances were used (see Table 1).

Sample denomination	Feeding distance [mm]	Spraying distance [mm]
40-350	40	340
60-425	60	425
80-500	80	500

Table 1 – List of WSP alumina samples according to feeding (FD) and spraying distance (SD).

The coatings were 1100 to 1300 μm thick and they were removed from the substrate after deposition in order to provide free-standing coatings and access to both free-surface and substrate-coating interface side of the coating. The interface surface (on the coating-substrate interface) and the top surface were polished; the coating was also cut and polished on the cross-section.

The results obtained on cross-sectioned samples are shown in Fig. 2. The figures contain results from both NHT and MHT. Despite a certain inconsistency at depths covered by both NHT and MHT instruments (~1,5 μm), the curves for all samples follow a very similar trend: rapid decrease of both hardness and elastic modulus at low depths and stabilization of the hardness and elastic modulus values above 2 μm depth.



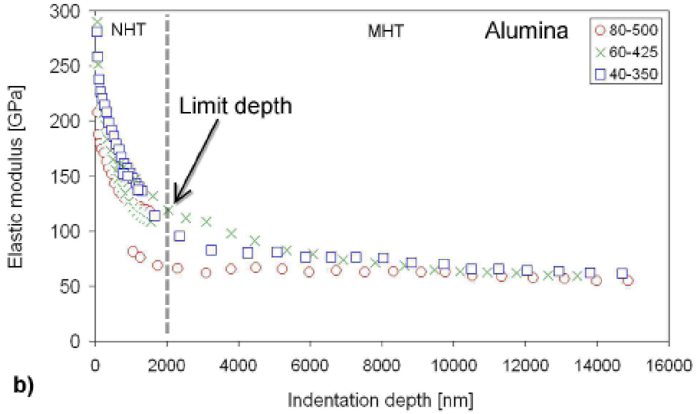


Fig. 2 – Hardness (a) and elastic modulus (b) as a function of indentation depth measured on cross-section of alumina coatings. For clarity reasons the error bars were omitted.

As there was a significant difference between the materials behavior below and above 2 μm, the depth of 2 μm was considered the 'limit depth'. Above this depth the influence of load on the mechanical properties of plasma sprayed coatings can be considered as negligible. This result was confirmed also for other plasma and HVOF sprayed ceramics and cermets. Outcomes of a larger experimental study presented at the 4RIPT conference in Lille (France, 2009) are now being reviewed for publication in a scientific journal.

The differences between coatings sprayed with different parameters were observed mainly at low loads (see Fig. 3) where hardness of the 80-500 was the highest, followed by the 60-425 coating and the 40-350 coating. Though the differences in the microstructure between the coatings were quite small [1], the indentation method revealed slightly different mechanical properties of each coating at low loads. These differences were very likely due to the different local temperature history of the deposited layers. Such measurements of mechanical properties would be very difficult to perform with the conventional Vickers microhardness as the indents would be very small and the results would contain rather large errors.

The indentation on the polished top- and interface surface was also done. The previously described decrease of the hardness and elastic modulus with increasing depth was observed in most of the measurements. In general, both the hardness and elastic modulus were higher at the top interface than at the substrate-coating interface. However, even despite careful polishing, the surface was often (due to the brittle nature of alumina splats and frequent pull-outs) so rough that only a few results could be successfully evaluated. This applied even to high load indentation up to 10 N and very likely similar problems would occur with conventional Vickers microhardness measurements.

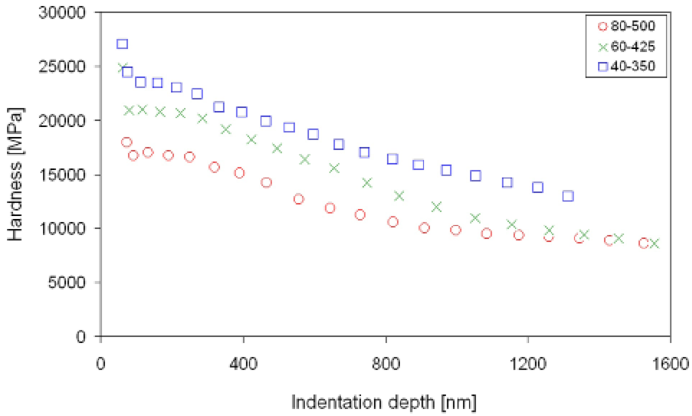


Fig. 3 – Hardness for the alumina coatings as a function of indentation depth. Error bars are omitted for clarity.

//// Plasma sprayed stainless steel

Stainless steel coatings were selected to represent a different type of materials to the plasma sprayed ceramics. The steel coatings were sprayed using the WSP torch from a 316L stainless steel powder onto a steel substrate. The thickness of the coatings was ~ 1300 μm except for sample 1412 whose thickness was 900 μm. The samples were sprayed under different conditions to achieve different fraction of oxides (Table 2) which are typical for metallic coatings sprayed in air.

	Sample denomination			
	1408	**1410**	**1411**	**1412**
Oxides content [%]	2.72	4.25	4.16	7.65

Table 2 – Oxide content in the 316L steel coatings.

The indentation was done with the CSM Instruments Nanoindentation Tester (NHT) at loads from 1 mN up to 100 mN with 20 cycles using the CMC method. Further, selected homogeneous areas were tested by single load indentations at 10 mN. All indentations were done with a Berkovich indenter.

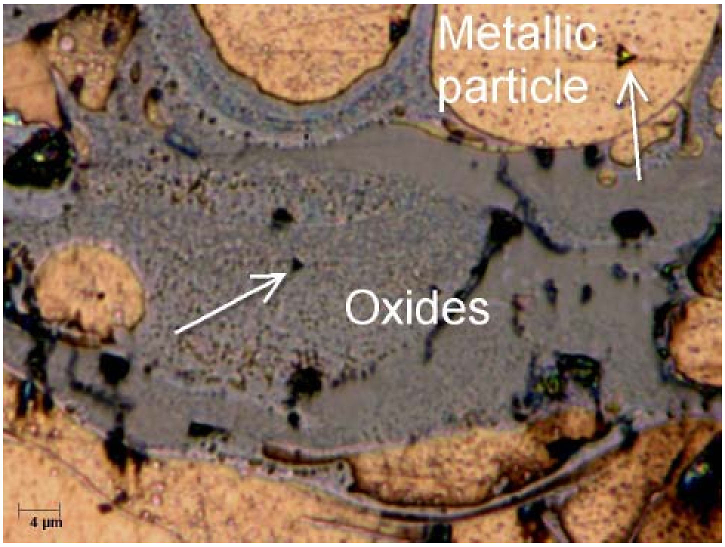


Fig. 4 – Precise location of indentations on WSP® sprayed steel (100 mN maximum load). Arrows show the indents.

In the case of the 1408, 1410 and 1411 coatings the oxides formed a rather thin layer around the metallic splats and measurement of the properties of the oxides would be strongly influenced by the surrounding metallic splats. Therefore indentation on metallic splats only was performed. On sample 1412 however, the fraction of oxides was sufficiently high and single indentations could be carried out on both metallic and oxide particles (Fig. 4). The prevailing metallic particles in all coatings were easy to target for both cyclic indentation and single load indentations. The chart in Fig. 5 shows the results of single indentation at 10 mN for all steel samples.

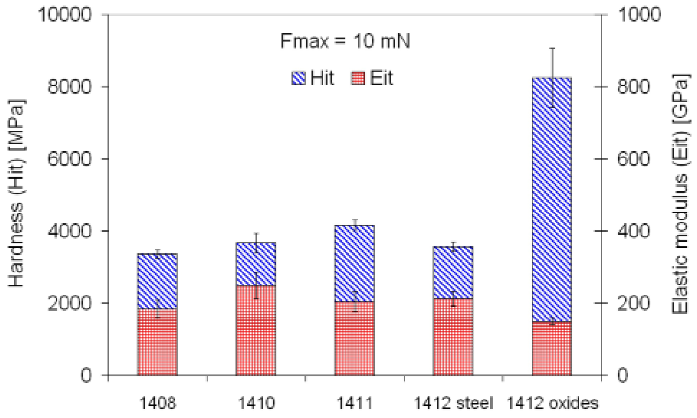


Fig. 5 – Hardness and elastic modulus of the steel coatings. Note high hardness of the oxides in sample 1412.

There was little variation in hardness and elastic modulus of all coatings observed; elastic modulus of all metallic splats was close to 200 GPa, which is the known value for bulk steel. Low load indentation can therefore effectively measure single phases or constituents in heterogeneous material such as thermal spray coating. Oxide particles on sample 1412 were clearly visible – though still relatively small – and their hardness was much higher than that of the surrounding metallic splats. On the other hand, the elastic modulus of the oxides (~150 GPa) is surprisingly lower than that of steel (~200 GPa). This could be explained by internal porosity of the oxide particles or intra-particular cracking [2] though a more detailed study would be required for proper understanding of this result.

The cyclic mode indentation showed that there was little decrease of neither hardness nor elastic modulus with increasing indentation depth (Fig. 6). This finding proves different mechanical behavior of the metal-based coating where the formation of the intrasplat cracks was suppressed due to the possibility of evolution of plastic deformation.

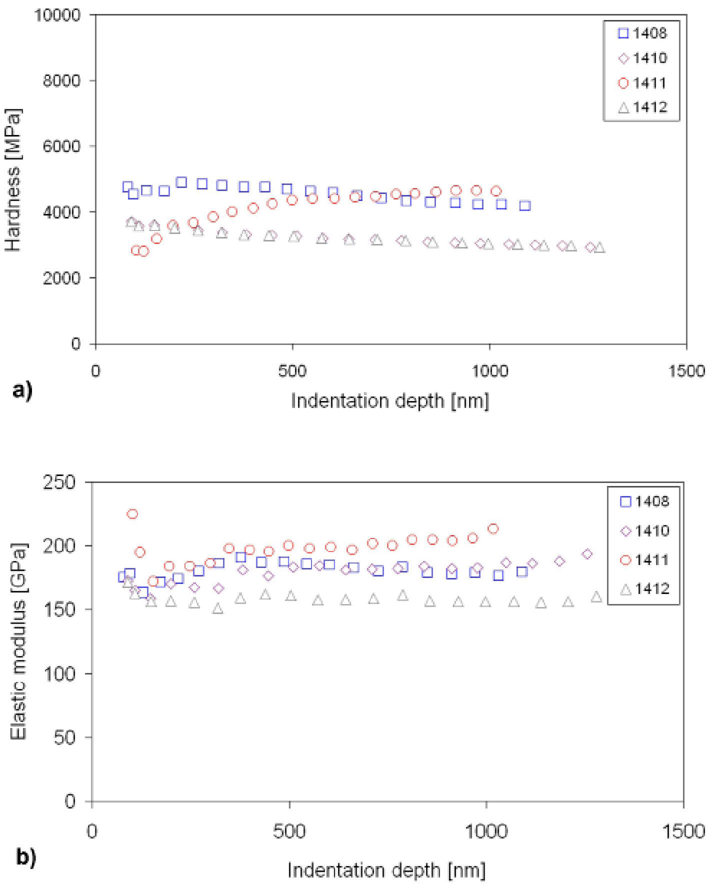


Fig. 6 – Hardness (a) and elastic modulus (b) of steel coatings as a function of indentation depth, each data point is mean value from about 5 indents.

The elastic modulus measurements by both cyclic and single load instrumented indentation are in very good agreement with the known values of elastic modulus of stainless steel. This confirms that the elastic modulus obtained by instrumented indentation corresponds to that obtained by other methods such as four point bending or tensile tests.

//// Indentation hardness dependence: force or indentation depth?

As mentioned above, in the CSM Instruments Application bulletin 29 and also elsewhere, hardness and elastic modulus of thermal sprayed coatings often depend on the indentation load. It is due to the heterogeneity of their structure, where different phenomena are involved when different volumes are tested. Many people report mechanical properties at a given loads. However, we suggest that rather than load the indentation depth should be cited when hardness or elastic modulus values are reported. The indentation depth can be easily related to the dimensions of the structural units of the coating and the variations of mechanical properties can be more easily explained and predicted. A plot showing dependence of depth on indentation load can be included in the result to link the depth-related data to load.

//// Conclusions

This Application Bulletin reports on the application of instrumented indentation over a large range of loads on plasma sprayed ceramic and metal coatings. It shows the advantages of this method on heterogeneous materials and allows both a detailed study of individual coating constituents and overall coating's properties which was not previously possible. The most important advantage of instrumented indentation for thermal spray coatings is its ability to make localized measurements: targeted testing of small volumes can reveal important material properties which can help to understand large scale (bulk) phenomena such as cracking and wear, originating from small structural units.

//// Acknowledgements

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//// References

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2. Musalek, R. Kovarik, O. Matejicek, J.: In-situ observation of crack propagation in thermally sprayed coatings. Accepted for publication in Surface and Coatings Technology, 2010.

CSM Instruments

This Applications Bulletin is published quarterly and features interesting studies, new developments and other applications for our full range of mechanical surface testing instruments.

Editor Dr Jiri NOHAVA

Should you require further information, please contact:

CSM Instruments
Rue de la Gare 4
CH-2034 Peseux
Switzerland

Tel: + 41 32 557 5600
Fax: +41 32 557 5610
info@csm-instruments.com
www.csm-instruments.com

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