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Application of resonant ultrasound spectroscopy to determine elastic constants of plasma-sprayed coatings with high internal friction

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ABSTRACT

We present a novel modification of resonant ultrasound spectroscopy (RUS) for analysis of elastic constants of plasma-sprayed coatings in the 'as sprayed' state, i.e. without removing the substrate. This modification is suitable for coatings with high internal friction ($Q^{-1} \gtrsim 10^{-2}$), which cannot be measured by RUS in the free-standing state due to strong damping. In combination with through-transmission measurements, this modification is able to provide full anisotropic tensor of elastic constants of the coating. As an illustrative example, the proposed methodology is applied to two steel coatings prepared by water-stabilized plasma (WSP) spraying. In addition to the moduli, also the internal friction parameters of the coatings are obtained. The results of RUS are compared to moduli determined by four-point bending tests, and it is shown that the anisotropic elasticity of both examined materials exhibits an elliptic form of transversal isotropy with four independent elastic coefficients.

1. Introduction

Elastic moduli are among the key parameters of surface coatings prepared by thermal spraying. Not only they are direct indicators of porosity and integrity of the coating, i.e. of the quality of mutual bonding of the splats [1,2], which makes them usable for adjustment of technological parameters of the spraying. Their exact knowledge is also essential for estimation of residual stresses arising due to quenching of the droplets and thermal expansion mismatch between the substrate and the coating [3–5], or stresses induced by elastic straining of the underlying substrate. From the latter point of view, the in-plane elastic constants are of the highest importance, as the mechanical loads imposed into the surface layer by the strain field of the substrate are, providing that the thickness of the coating is small, predominantly of in-plane nature. Lastly, the elastic moduli of the coating are basic input parameters necessary for numerical (e.g. FEM) modeling of coated bodies and their reliable knowledge is for such computations substantial.

For plasma sprayed coatings, the elastic moduli are most usually determined by micro- or nano-indentation [6–8]. This method has many advantages, providing for example some insight into the anisotropy of the coating [2,6]; the nano-indentation can be also used for mapping of the moduli across the coating microstructure [9]. On the other hand, the stresses applied on the coating during the indentation are locally very high, usually above the yield limit of the material but limiting to zero with increasing distance from the indentation point. Hence, due to the well-known strongly non-linear character of the elastic behavior of the plasma-sprayed coatings [10], it is not exactly clear which stress level the obtained elastic constants correspond to. Another undesirable feature of the indentation method is the strong dependence of the measured elastic modulus on the indentation depth and load-influenced volume [7,11].

For these reasons, alternative methods working with lower and better defined stress levels are often employed, ranging from classical (static and dynamic) tensile and bending tests [12–15] to neutron scattering [16], X-ray diffraction [17] and ultrasonic methods. A wide variety of ultrasonic techniques has been successfully applied for this purpose so far, including contact measurements of velocities of bulk and surface acoustic waves [18–20], immersion methods [21], laser-based ultrasound [22] and Brillouin scattering [23]. The main motivation for the use of these methods is that the level of strain oscillations carried by the ultrasonic waves is extremely low ($\sim 10^{-6}$, which is e.g. by two orders of magnitude lower than in typical four-point bending tests), so a purely linear elastic response is obtained without inducing any damage to the material.

Most recently, Tan et al. [24] have applied resonant ultrasound spectroscopy (RUS, [25,26]) to determine elastic constants of free-standing coatings prepared by atmospheric plasma spraying (APS) and high-velocity oxygen-fuel (HVOF)process. RUS is an advanced ultrasonic method based on determination of the elastic moduli from resonant spectra of free elastic vibrations of a small sample of the examined material. This approach seems to be very promising,

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since the RUS method works with wavelengths comparable to the dimensions of the samples (few millimeters), which are incomparably larger than any characteristic length-scales of the microstructure (dimensions of splats, pores and cracks). The resulting elastic constants can be therefore expected to describe well the macroscopic (homogenized), purely elastic behavior of the coating. Moreover, if the used samples are thin plates (as in the case of Tan et al. [24]), the majority of the vibrational modes are bending modes, so the RUS measurement is particularly sensitive to the bending stiffnesses of the sample, which are closely related to the in-plane elastic moduli.

Nevertheless, the RUS method is applicable only for coatings with acceptably low internal friction, for which the sample can be set into resonance and individual resonant peaks in the spectrum can be easily distinguished. For some plasma sprayed coatings, however, the typical values of the internal friction are relatively high ($Q^{-1} \sim 10^{-2}$, [27]) due to their somehow loose microstructure that is desirable e.g. for enhanced strain and thermal shock tolerance of the coating. Such high value of Q^{-1} may result in problems both in generation of the vibrations (higher energy of the generating pulses is needed) and in identification of resonant peaks (the broadened peaks are overlapping and the signal-to-noise ratio is decreased). In this paper, we present a modification of RUS method for the analysis of supported coatings instead of the free-standing ones. In such case, i.e. if the sample for RUS consists of a thick substrate with a significantly thinner coating deposited on it, the resultant resonant spectrum is mainly the spectrum of the substrate, with the individual peaks only slightly shifted by the presence of the coating. Similarly, the high internal friction in the coating leads only to limited broadening of the individual resonant peaks, so the identification of the resonant frequencies is still possible, and the sample itself can be easily set into resonance.

This modified approach originates from classical RUS method for evaluation of thin layers [26,28], which is based on the comparison of the resonant spectra of the same substrate sample before and after deposition of the layer. The shifts of the peaks then carry information on the in-plane elasticity of the layer. In a similar manner, in this paper we obtain an information on the in-plane elasticity of the coating by analysis of the evolution of the spectrum of one sample during subsequent removal of the coating by grinding (as described in details in Section 3.1.). We show that such analysis is possible even if the spectra are significantly damped (internal friction of the coating $\gtrsim 10^{-2}$), and, when complemented by classical through-transmission measurements, this approach enables determination of the full anisotropic tensor of the elastic constants. Moreover, from the shifting and broadening of the peaks with increasing thickness of the coating, additional information on the coating can be extracted, which we employ in this paper for the determination of the internal friction parameters. In summary, the experimental technique proposed in this paper differs from the conventional RUS method in two points: (i) the measurements are carried out on heterogeneous, sandwich-like samples consisting of a substrate (known material with low internal friction) and thick layer of the coating (unknown, highly dissipative material); (ii) the measurements are repeated with several successive removals of the coating material, so the dependence of the resonant spectrum on the thickness of the coating is obtained and used for inverse calculation of the elastic constants of the coating.

A similar approach to the one presented in this paper was also developed by Lauwagie et al. [29], who determined in-plane moduli of air-plasma sprayed thermal barrier coatings (TBC) from resonant spectra of small bars including both the substrate material and the coatings. Lauwagie et al. [29] used a finite element code for the calculation of resonant frequencies of such samples and obtained the resulting elastic constants by tuning the input parameters of this code until an optimal fit was reached. Unlike the approach reported in [29], our method is comparative: it extracts the information on the elastic moduli of the coating from the differences between the spectra of the substrate with and without the coating. This ensures that our results are much less influenced by the uncertainties in the knowledge of the properties of the substrate itself. Moreover, fitting the whole evolution of the spectrum with successive removals of the surface layers enables a deeper insight into the structure of the coating, and, as also shown in this paper, the estimation of the effective location of the roughened substrate-coating interface. On the other hand, the method described in this paper is destructive: the coating is fully removed after the measurement.

2. Materials under study

Two stainless steel coatings with different volume fractions of oxides were chosen for the RUS analysis in order to observe if the proposed RUS method will show the expected difference in the stiffness of these coatings. Both these coatings were sprayed onto common low carbon steel substrates from a 316L powder of nominally 106–150 μm size range (Stamont International, Slovakia) using a water stabilized plasma (WSP) torch (Institute of Plasma Physics, ASCR, Czech Republic) with the following parameters used: torch power 160 kW, powder feeding distance 100 mm, and powder feed rate 270 g·min^{-1}. In both cases, the coating thickness was approximately 1 mm and the substrate thickness was 2.5 mm (lateral dimension 25 × 100 mm). The difference in the content of oxides was deliberately induced by different spraying distances (as a consequence of interaction of the flying molten droplets with a surrounding atmosphere): one of the coatings was deposited at distance of 300 mm, and the second at distance of 500 mm. Oxygen content in the coatings was determined by electron probe microanalysis (EPMA) in a Camscan 4DV scanning electron microscope with a Link AN1000 (Link Analytical, UK) analytical system. The volumetric percentage of the oxide phases was determined by image analysis of the SEM images of polished cross sections. The results were as follows: the coating deposited at 300 mm spraying distance contained 2.72 wt.% (11 vol.%) of oxides and will be hereafter referred to as the LO (low oxides) coating; the coating deposited at 500 mm spraying distance contained 7.65 wt.% (20 vol.%) of oxides and will be hereafter referred to as the HO (high oxides) coating. SEM micrographs of the coatings under study and of the substrate-coating interfaces are given in Fig. 1.

Preliminary estimations of the in-plane Young's moduli of these two coatings were obtained by four-point bending (4 PB) tests. This was done in an Instron 1362 universal testing machine (Instron, UK). The coatings were tested in the as sprayed condition (on substrates) and loaded in compression up to a small strain of 0.05% to minimize the structural damage. The substrate and coating contributions to the total stiffness were separated according to [30]. The result was that $E_{HO} = (45 \pm 9)$ GPa and $E_{LO} = (28 \pm 7)$ GPa, where the subscripts LO and HO correspond to the denotation of the respective coatings.

From each of the examined materials, a rectangular parallelepiped sample for the RUS measurements was cut, containing both the coating and the underlying substrate and with top and bottom faces parallel to the substrate-coating interface. As explained in the Introduction section, the applied modification of RUS required the coating to be significantly thinner than the substrate, which was not fulfilled for the initial thicknesses of the coatings. For this reason, a part of the coating was removed prior to the RUS measurements by grinding, until the ratio between the thickness of the substrate and of the coating higher than 5:1 (about 2 mm of the substrate to 400 μm of the coating) was reached. After this first partial removal of the coating, all faces of the samples were ground and polished to ensure plan parallelism of the opposing faces. Final dimensions of the two resulting samples are listed in Table 1 (in agreement with this table, we will from hereafter use the denotation a for the thickness of the sample in the direction perpendicular to the substrate-coating interface.)