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DVS – DEUTSCHER VERBAND FÜR SCHWEISSEN UND VERWANDTE VERFAHREN E.V.	**Guide to the production of microsections and the evaluation of thermally sprayed coatings**	Technical Bulletin DVS 2310-1

Reviewed and unaltered version of the December 2007 issue

Content:

1 Introduction

The Leaflet DVS 2310-1, 1984 issue, had to be revised because the results of a round robin test showed that the recommended preparation method for bonded systems with a ceramic layer did not produce a satisfactory result. The layers are therefore not prepared.

Another reason was due to the fact that the once preferred method of manual microsection preparation must be regarded as being qualitatively inadequate. Grinding and polishing processes must be carried out automatically or using suitable mechanised systems in order to be able to keep the preparation parameters contact pressure, speed and lubricant dosage constant and reproducible.

After completion of the round robin test, where the preparation for grinding was primarily based on the use of SiC paper, new grinding methods have been introduced recently under the consideration of newly designed diamond-tipped grinding discs with a different grain size, which are taken into account in this revised version in order to meet the state of the art requirements.

With regard to the requirements of the layer and its evaluation, there may be significant differences in the quality requirements since they are being used in different industries. The permissible irregularities determined during the materialographic examination for the respective application must be defined by the contracting parties with reference to the components.

With certain applications, roughness and micro-hardness measurements can be of help especially for comparative nominal-actual statements during the materialographic examination.

Thermally sprayed coatings can be metallic or ceramic or a combination of metallic and ceramic parts or plastic parts [1; 3; 4]. In practice, quality control is only possible to a limited extent using non-destructive test methods [2; 6]. Materialographic examinations can however be used to evaluate the characteristic features of a sprayed coating such as thickness, structural texture (structure, porosity), bond to base material and hardness (e.g. of individual phases).

The combination of hard and soft, brittle and ductile, wear-resistant and non wear-resistant materials or microstructure constituents (phases) in the sprayed coating/base material compound and/or within the layer requires some special characteristics to be observed during the materialographic production of microsections [8; 16; 17; 19; 20; 21].

The following work steps which describe the production of microsections give full details of the correct removal and microsection preparation of specimens with thermally sprayed coatings.

2 Work steps for the production of microsections

2.1 Sample selection

If an original part is not available for the destructive test, then a material coated under the same spraying conditions and movement sequences can be examined for which the base and coating material and the coating thickness should correspond as closely as possible to the original. The objective is to aim for a specimen temperature which is comparative to that of the component when coating.

Common specimens for a sample coating comparative to the original:

- Solid round specimen [5], diameter 40 ± 0.1 mm, coated on the peripheral or face side.
- Round pipe specimen, outer diameter 40 mm, wall thickness 2 mm.
- Flat specimen, recommended dimension l x w x h: 100 x 15 x 1.5 (values in mm).

2.2 Sampling

Sampling is crucial to the correct evaluation of the sprayed coating. Samples should be taken by means of wet separation in the direction of the cut to the base material. With round specimens, only sector 1 is useful to the evaluation, Figure 1.

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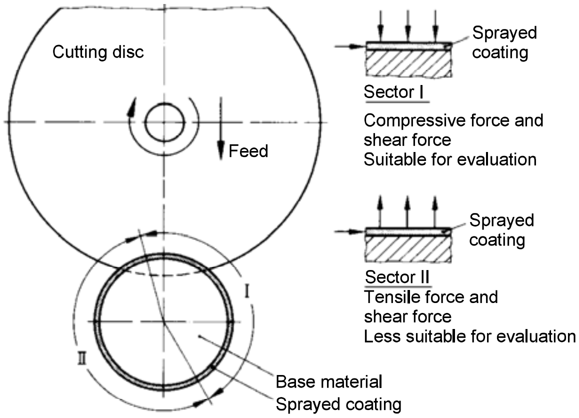

Figure 1. Sampling.

If the component size allows it, extremely brittle layers can under certain circumstances be infiltrated with a hardenable synthetic resin before cutting to protect the microsection specimen against edge chipping or spalling during the cutting process. Failing that, the test specimen should be separated roughly first and if necessary infiltrated and then separated again under vacuum.

For **metallic** materials, you can use thin, rubber or synthetic-resin-bonded aluminium oxide or silicon carbide cutting discs. Maximum peripheral speed of the cutting discs 45 m/s.

The following usually applies:

– Hard materials: soft cutting discs
– Soft materials: hard cutting discs

Ceramic materials must preferably be cut with synthetic-resin-bonded diamond-tipped discs. Other cutting discs cause more severe breakouts which calls for a lot of pregrinding to remove them. Maximum peripheral speed of the cutting discs: 15 m/s. The manufacturers of cutting discs recommend specific discs for the respective application.

The mechanical load and overheating of the sprayed coating during cutting must be avoided at all times. Working at a constant feed as opposed to a constant pressure is preferable.

2.3 Identification

The separated specimens can be identified permanently through engraving on the back; the marking must be transferred to the bezel.

2.4 Embedding

All coated specimens must be embedded [10; 11]. Even if semi-automatic grinding and polishing processes are used, embedding is still necessary in order to simplify clamping in the specimen holder. The most important embedding methods are hot and cold embedding in synthetic resins.

Hot **embedding** takes place in heatable embedding presses in which the specimens are heated together with the embedding agent and whilst at the same being loaded with a constant pressure. Hardening temperature and pressure must be selected according to the manufacturer's specifications. With hot embedding, sensitive layers may become damaged. It should therefore preferably be used for metallic layers.

For **cold embedding,** hardenable synthetic resins are available. To obtain microsections with a good marginal sharpness for the examination, embedding agents with the lowest shrinkage tendency must be selected to prevent a gap from forming between the embedding agent and specimen [10; 11]. For the same reason, cold and hot embedding agents can be supplied with filler materials to assimilate the different hardness and abrasion behaviour between the synthetic resin and specimen within specific limits, and to prevent the edges of specimens from rounding.

In order to subject the composite material to as low a thermal and mechanical load as possible for the examination, in particular where layers are extremely porous and to prevent breakouts in the layers, we recommend embedding in vacuum in cold hardenable plastics. Ceramic materials should therefore generally be embedded cold.

Very thin layers (e.g. < 10 µm) or thin layer areas (e.g. diffusion zones) call for an angle microsection to enlarge the area [12] and/or the electroless coating in advance with copper or nickel, whereby the marginal sharpness and contrast between the layer and embedding agent are improved.

2.5 Grinding and lapping

Reproducible preparation results can only be achieved if the grinding and polishing processes are semi- or fully-automated since the necessary constancy of the contact pressure cannot be achieved manually [17; 18; 21].

Wet grinding with a bonded or loose grinding grain (e.g. water resistant silicon carbide papers, diamond grinding pads, diamond suspensions) is commonly used. With this machining process, material is separated from the sanded back surface. At the same time, the areas close to the surface are deformed. The entire depth of the surface layer which is disrupted by the roughness (depth of scratch) and deformation is dependent on the grain size of the grinding agent, contact pressure when grinding and the hardness of the substrate and coating material, Figure 2.

Coarse and fine granulations must be removed from the separation damage to the layer and the disrupted surface layer, see Tables 1.1; 1.3; 2.1a; 2.1b; 2.3a; 2.3b. Changing to a finer grain size can only occur if no further grooves are detected in the previous grain size. Additional grinding does not improve the quality of the sanded back surface.

If, in exceptional cases, grinding needs to be done by hand, the specimen when it is changed, must be offset by 90° to the next finest grain size.

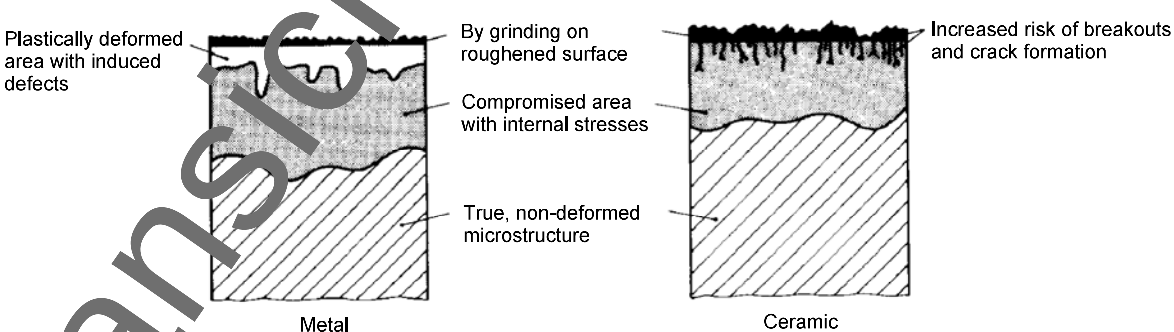

Figure 2. Surface defects in metal and ceramic layers [8].