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Structure and Properties of PVD TiB_2 Coatings

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For optimizing machine parts about their function and lifetime not only the design has been varied but also the materials. Where moving parts are in contact with each other, mostly only a few nanometer thick layer guarantees the function. With surface coating by PVD the properties and structure of this layer can be modified, so the use of bulk material is not necessary. This study is about the development of a hard, wear resisting TiB_2 coating for lubricant-free roller bearings. Therefore several pretensions must be fulfilled, for example, no change in the surface topography of the raceways and low temperature coating process for tempered materials. Consequently all coatings were done with the Magnetron sputter ion plating (MSIP) process. For the target material a hot isostatic pressed titanium diboride plate was used. This target is electrically conductive, so that the sputtering could be done with a dc plasma. Three different substrate materials were examined. These were tempered bearing steel (100 Cr 6), silicon nitride (Si_3N_4), and a cutting tool material (HS 6-5-2). For optimizing the coating process and adapting it to the different materials, the temperature and the bias voltage were varied. While Si_3N_4 and HS 6-5-2 are insensitive to the coating temperature, the temperature of the tempered 100 Cr 6 must be lower than its tempering temperature otherwise a reduction in the hardness cannot be excluded and the support of the coating is not sufficient. The coatings were characterized by their microstructure and their mechanical properties. © 1997 Academic Press

INTRODUCTION

In the past few years several hard coatings have been deposited on wear loaded machine parts or cutting tools by physical vapor deposition (PVD). The deposition of TiN, CrN, TiAlN, and TiC is state of the art. These coatings are all carbides or nitrides. Another group of hard materials, the borides, have no significant part in commercial coatings.

This study examines selected properties of titanium diboride coatings and their dependence on the coating parameters substrate temperature and bias voltage. All coatings were deposited with the magnetron sputter ion plating (MSIP) process. Three groups of substrate materials were coated: tempered 100 Cr 6 as an example for temperature-sensitive metals, silicon nitride for ceramics, and HS 6-5-2 for temperature-insensitive cutting steel.

The coatings were characterized by their XRD structure, their rupture structure, their microhardness, and their critical load.

SUBSTRATE AND TARGET MATERIALS

The bearing steel 100 Cr 6 is often used for wear applications at room temperature up to 100°C. The material can be hardened up to 63 HRC. The examined 100 Cr 6 was quenched and tempered to 60 HRC. Its tempering temperature is 180°C. Therefore the substrate temperature during the coating process has to be lower than 180°C. Otherwise a reduction of the substrate hardness cannot be excluded and the support of the coating is not sufficient.

Another examined substrate material was silicon nitride. Specimens were produced in a sintering process. Its microhardness is about 1800 HV 0.05. The maximum coating process temperature of 500°C has no effect on that material, so all coating parameters could be varied in a wide range.

The third substrate material that was coated was a cutting steel (HS 6-5-2). This material was quenched and tempered to a hardness of 62 HRC. Its tempering temperature is 550°C. This temperature was not reached in the coating process, so no restrictions for the coating parameters were taken into consideration.

A hot isostatic pressed titanium diboride plate was used as a target for the MSIP process. Its diameter is 75 mm and is 5 mm thick. TiB_2 is electrically conductive, so sputtering with dc plasma is possible.

COATING PROCESS

All coatings were deposited in a modified Z400 MSIP plant, Leybold AG. The MSIP process is principally outlined in Fig. 1.

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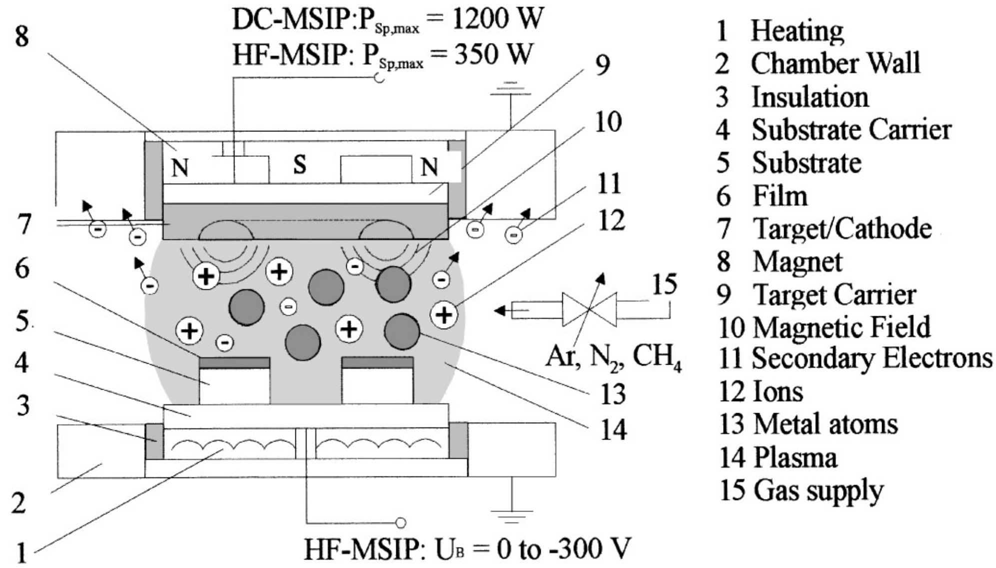

FIG. 1. The MSIP coating process.

The varied coating parameters were the bias voltage and the substrate temperature. At all coatings the argon pressure was 1 Pa and no reactive gas was added. The dc plasma power was constantly 400 W. Before the deposition was started the specimens were cleaned in a plasma etching process. The variation of the bias voltage and the substrate temperature are shown in Figs. 2 and 3.

The 100 Cr 6 specimens 4 and 5 had partial delaminations of the coating. No further examinations of these specimens were possible. Specimens 3, 4, and 5 of silicon nitride and HS 6-5-2 had only a powderlike coating, so also no further examinations of these specimens were possible.

RESULTS

All remaining coatings were examined with computer-assisted XRD (Bragg–Brentano). A Co tube was used for X-ray generation. The crystalline structure was scanned in a step-scan mode where tube and detector were moved with an angle relationship of 1 : 1 while the specimen was fixed. In all cases, independent of substrate material and coating parameters, the XRD plots showed only the substrate peaks. Figures 4, 5, and 6, the XRD plots of coated and uncoated 100 Cr 6, HS 6-5-2, and Si_3N_4 substrates, respectively.

All coatings showed no significant rupture structure. Figure 7 shows the rupture structure of a coated 100 Cr 6 substrate. The coatings were compact and homogeneous. The surface of the substrate was exactly reproduced. Effects of substrate material or coating parameters were not detectable.

Figure 8 gives an overview of the critical load and the microhardness of the different coatings on silicon nitride and HS 6-5-2.

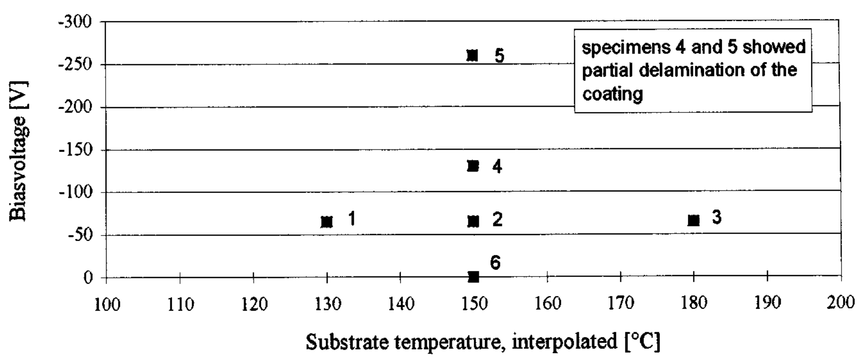

FIG. 2. Deposition parameters on substrate material 100 Cr 6.

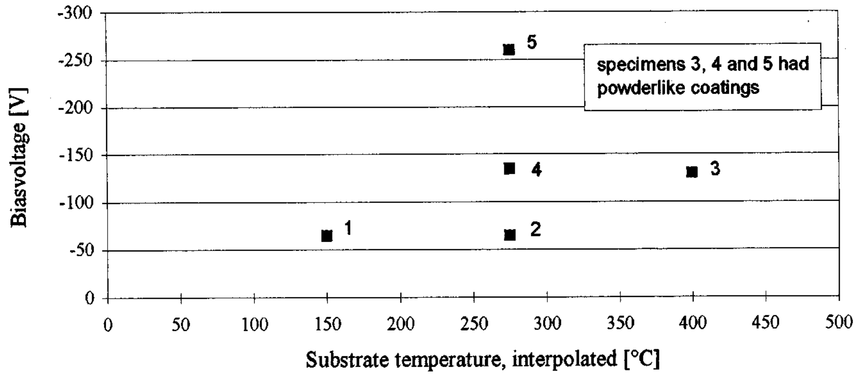

FIG. 3. Deposition parameters on substrate materials Si_3N_4 and HS 6-5-2.

The critical loads are constant when the deposition parameters are varied, but are dependent on the substrate material. On HS 6-5-2 specimens, 30 N load is achieved, while on Si_3N_4, 20 N were measured. The highest microhardness of 3000 HV 0.05, respectively 3200 HV 0.05, is achieved by deposition with bias voltage − 65 V and substrate temperature 150°C.

The coatings on 100 Cr 6 had the highest microhardness with deposition without any bias voltage and substrate temperature 150°C (see Fig. 9). But only critical loads in the range of 10 to 18 N were measured. The higher value is achieved by deposition without external heating and bias voltage − 65 V. The substrate hardness of 60 HRC was reduced to 56 HRC at deposition with − 65 V bias voltage and substrate temperature about 180°C.

For wear resistant applications a high adhesion between coating and substrate is necessary. Therefore a thin interlayer of titanium was deposited on 100 Cr 6. The structure of the titanium was columnar (see Fig. 10). Normally this structure reduces the properties in comparison to a dense and homogeneous structure. In this case the microhardness of the cover layer was not reduced but the critical load was increased to 68 N. The effect of metallic interlayers at the substrate materials silicon nitride and HS 6-5-2 has not been examined yet.

CONCLUSIONS

The deposition of titanium diboride on several different substrate materials with MSIP is possible. The TiB_2 target can be sputtered with a dc plasma. For optimizing the process the parameter bias voltage and substrate temperature have been varied. The XRD, rupture structure, microhardness, substrate hardness, and critical load were examined.

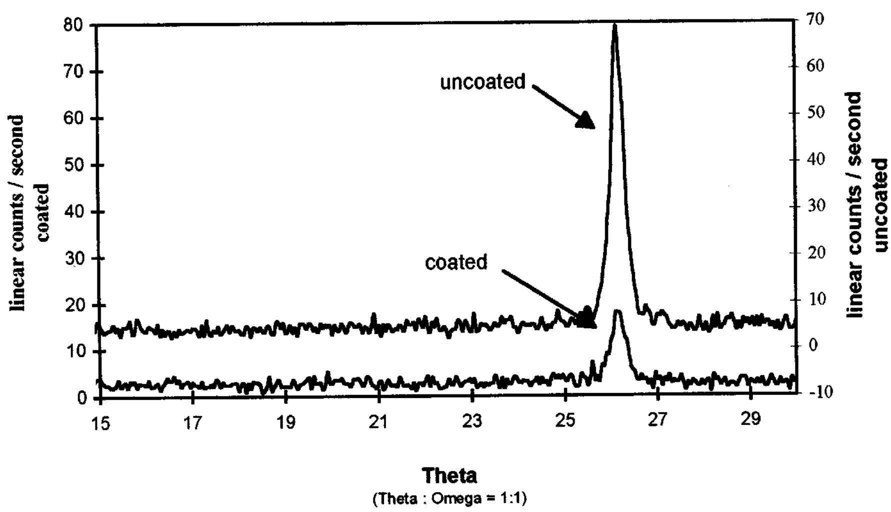

FIG. 4. XRD plot of 100 Cr 6 specimens, coated and uncoated.

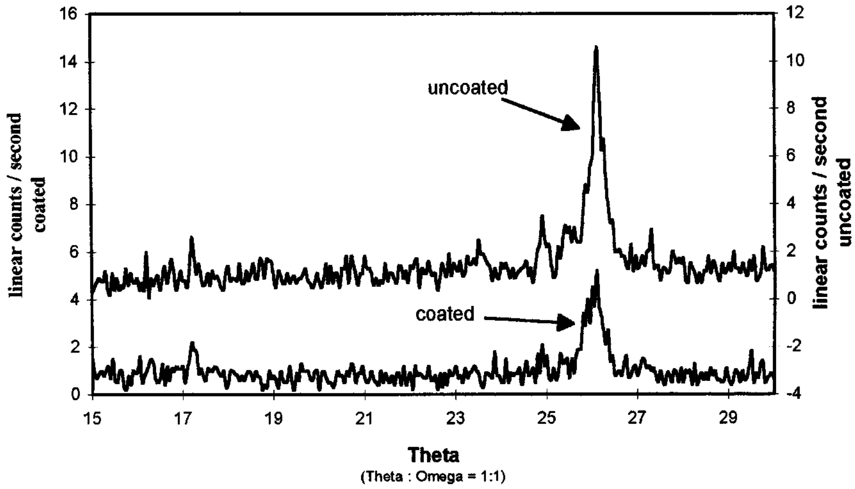

FIG. 5. XRD plot of HS 6-5-2 specimens, coated and uncoated.

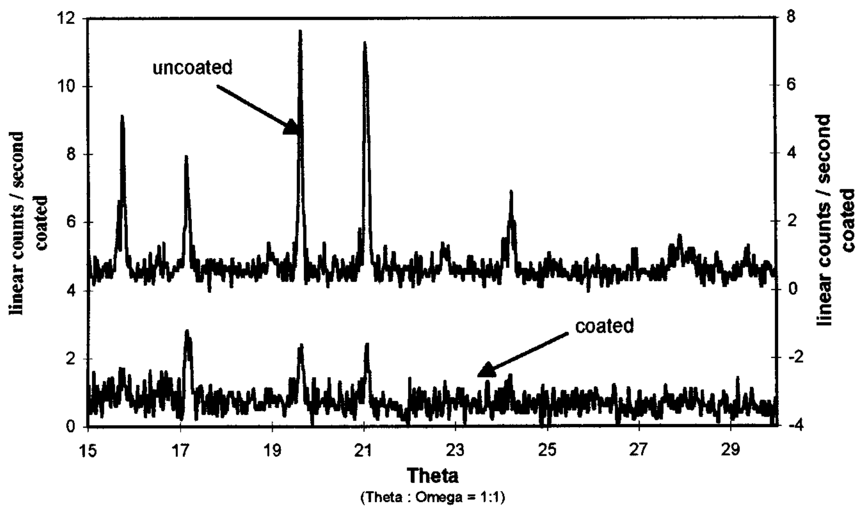

FIG. 6. XRD plot of Si_3N_4 specimens, coated and uncoated.

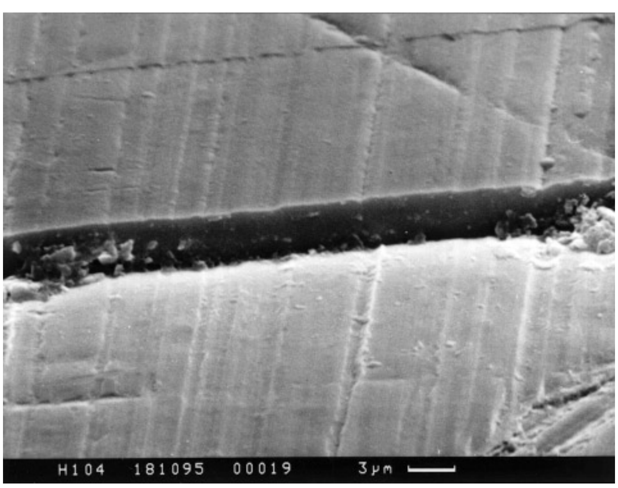

FIG. 7. Rupture structure of TiB_2 coating on 100 Cr 6.

Delamination, respectively powderlike, coatings were detected when the coatings were deposited with bias voltages above -65 V and high substrate temperatures. In their properties the examined coatings were independent of substrate material and deposition parameters mostly. Only the microhardness is influenced by the bias voltage and the substrate temperature. The highest microhardnesses were achieved by deposition with low bias voltage and low external heating. These parameters need only little modification for adapting the coating process to different substrate materials.

These parameters make the coating of thermal sensible materials with hard titanium diboride possible. The only restriction is that, for example, at tempered steel, the temperature during the coating process must be lower than the tempering temperature. By changing the plasma power and

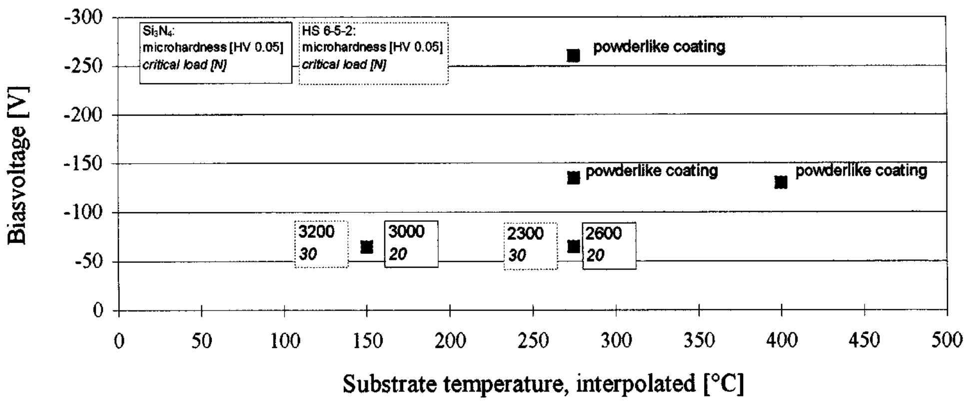

FIG. 8. Microhardness and critical load of coated silicon nitride and HS 6-5-2 samples.

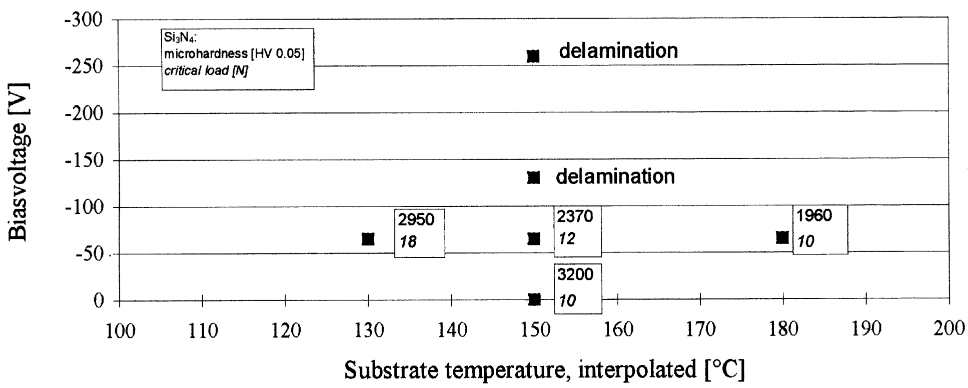

FIG. 9. Microhardness and critical load of TiB_2 coatings on 100 Cr 6.

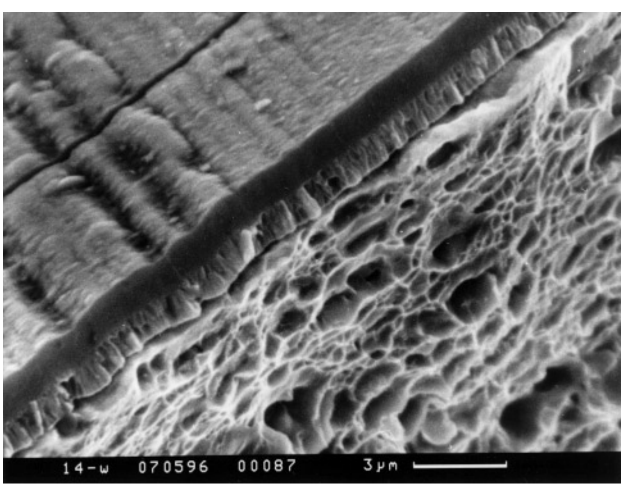

FIG. 10. Rupture structure of TiB_2 coating on 100 Cr 6 with a titanium interlayer.

the geometry of target and substrates the minimum coating temperature can be decreased below 100°C.

The critical load of coated 100 Cr 6 substrate can be increased by deposition of a thin titanium interlayer. The microhardness of the titanium diboride coating is 3200 HV 0.05 while a critical load of 68 N is achieved. With these deposition characteristics and properties titanium diboride is an excellent coating, if a hard, exactly reproduced surface is needed and the substrate material is temperature sensitive.