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Metallization of Thin Al_2O_3 Layers in Power Electronics Using Cold Gas Spraying

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A successful combination of insulating substrates with conducting metal coatings produced by cold spraying could open new industrial application areas like the fabrication of power electronic components. For minimizing the number of industrial process steps, insulating ceramic layers should ideally be processed by thermal spray techniques. Thus, this study investigates the impact behavior and coating formation of ductile metallic feedstock powders onto brittle ceramic coatings. With respect to high electrical conductivity of the metallic lines and good electrical insulation of the ceramic interlayer, copper was cold gas sprayed on previously thermally sprayed Al_2O_3 coatings. Successful cold coating formation requires different strategies for the activation of the ceramic layer to increase adhesion and to avoid brittle failure. These both can be achieved either by applying a bondcoat on the ceramic layer or using heated substrates during the cold spray process.

Keywords alumina, brittle substrate, cold gas spraying, copper, electronic application, surface activation

1. Introduction

Cold gas spraying enables the deposition of metallic coatings with very low oxygen content and therefore high electrical and thermal conductivity (Ref 1). In the past, this technique was extensively applied to coat metallic substrates. Fabrication of metallic layers on brittle substrates like ceramics is still a challenge, but could open new industrial application areas for this relatively young spraying technology.

One idea for a concrete application is the assembly of a high power electronic module as shown in Fig. 1. Conventional modules consist of a heat sink, e.g., a copper cooling block, onto which a Direct Copper Bonding (DCB) base plate is soldered. On top of the base plate, the electronic device is attached. Such a high power electronic module has to process currents of several hundred amperes. As a consequence, thermal stresses created inside of the solder connections lead to a reduction in the lifetime of the device. A new production concept could eliminate the solder connections by replacing the DCB by a combination of (i) thermally sprayed, electrically insulating Al_2O_3 and (ii) cold-sprayed, high conductivity Cu layers.

The bonding mechanism between metals in cold gas spraying is well understood (Ref 2-4) and is attributed to the extreme plastic deformation of both the particle and the substrate, leading to the occurrence of shear instabilities. However, brittle substrates like ceramics will not undergo severe plastic deformation but are likely to break instead. So far, only a very few attempts have been made to apply cold spraying on brittle and hard substrates (Ref 5-8).

Successful bonding in cold spraying is described by the ''window of deposition'' (Ref 3). With respect to mechanical and thermal properties of the feedstock material, it defines combinations of particle impact temperature and particle impact velocity, which have to be met by impact conditions of the particle onto the substrate (which is assumed to be of the same material).

However, the combination of plastically deformable and rather rigid and brittle material during cold spraying requires that most of the impact energy is converted into the local deformation and heating of the ductile component. Thus, using composites or spraying metals on ceramic surfaces would result in more severe deformation of the ductile particles. Moreover, time scales for deceleration get shorter, causing locally higher stress levels at the interfaces than for impacts on softer material. In particular, these locally high stress levels could cause failure of brittle substrates. Therefore, brittleness of ceramics as substrate or as intermediate layer will likely narrow the ''window of deposition,'' as sketched in Fig. 2. The range, where the amount and shapes of local shear instabilities at surfaces of impacting particles are sufficient for bonding, but also exclude failure of the ceramics, is dependent on

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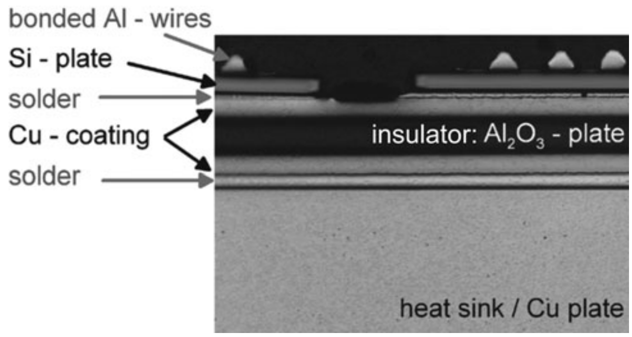

Fig. 1 Cross section of a conventional high power electronic module (OM micrograph)

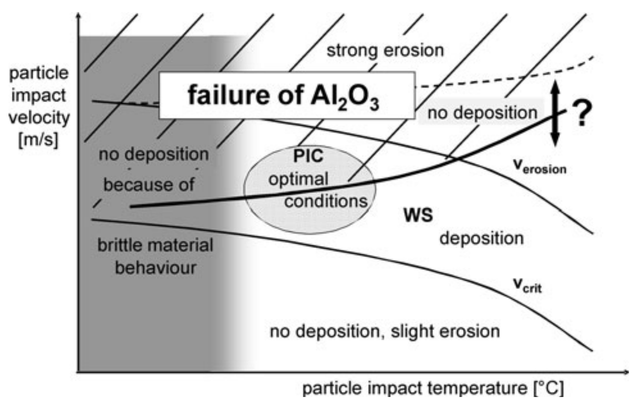

Fig. 2 Schematic of the "window of deposition" of Cu (WS: window of sprayability, PIC: regime of particle impact conditions) (Ref 3). For cold spraying on ceramics, the range of successful coating formation is narrowed by a possible failure of Al_2O_3 layers under local mechanical loads. The critical momentum for failure should be dependent on locally attained temperatures, as additionally sketched in the graph

the mechanical properties of both. In particular, the ceramic material should withstand high demands. So far, thermally sprayed ceramic coatings were mainly investigated under quasi-static loads (Ref 9, 10). Thus, the lack of systematic approaches of ceramic coatings under high strain rates necessitates experimental optimization of cold spray conditions for metals on ceramic layers.

In thermal spraying, different attempts are known from the literature to improve adhesion. Surface activation plays a major role in the successful joining process of a variety of material combinations. The use of interlayers as bond coats is common in thermal spraying to improve the bond strength of coatings (Ref 11). Bond coats are likely to adhere to the substrate because of diffusive interactions at the interface and forming of strong chemical bonds. For cold gas spraying of metals onto Al_2O_3 substrates, interdiffusion should not occur because of the short time scales. Nevertheless, chemical bonds over interfaces of different material layers could form. For such activation of Al_2O_3, aluminum is a promising candidate to act as a bond coat because of the chemical affinity to oxygen (see section 3.2) and is used as one possible surface activation method in this study.

Another attempt to improve adhesion is heating of the substrate during spraying. Investigations of the splat formation in thermal spraying have revealed a correlation of splat shape and therefore adhesion to the substrate temperature; see for example Ref 12-22. According to these results, increased substrate temperatures usually enhance disk-shaped splats, which are identified as a precondition for a good adhesion of the sprayed coating (summarized in Ref 15). Corresponding adhesion tests of coatings sprayed on heated substrates show therefore a clear tendency to higher bond strengths as compared to cold substrates (Ref 23-25). One reasonable explanation for this effect is the desorption of adsorbates or condensates from the surface at higher temperatures. This explanation is supported by experiments carried out in low-pressure atmospheres: At lower pressures, the shapes of the thermal-sprayed splats change from splashed to disk-shaped. This observation is explained in terms of evaporation of surface adsorbates or condensates at pressures below the vapor pressure of the substances (Ref 20). For cold gas spraying, the effect of substrate temperature has barely been investigated so far, and only a very few results for metals sprayed on metal substrates can be found in the literature (Ref 26-28). For aluminum sprayed on steel (Ref 27) and copper sprayed on stainless steel and aluminum (Ref 28), it was observed, that a higher substrate temperature could increase the deposition efficiency or even enable build-up of coatings. Although the physical background of these observations could not be clarified yet, higher substrate temperatures during the spraying process seem to support the adhesion in general. Consequently, heating of the ceramic substrate during the cold spray process is used alternatively to the surface activation via bond coats.

The objectives of this research study are therefore to optimize cold spray conditions to produce well-bonded metal coatings on thermally sprayed Al_2O_3 layers. This optimization includes the usage of different surface activation methods, namely an aluminum interlayer as bond coat or heating of the substrate during the spray process. Both these alternatives will be presented and discussed.

2. Experimental Procedures

The ceramic coatings should be electrically insulating and provide sufficient mechanical stability in power electronic applications. Both these requirements should be met by thermally sprayed Al_2O_3 coatings with low porosity and good adhesion to the substrate. With regard to the application requirements, Al_2O_3 coatings should have a thickness of about 200 μm. The mechanical stability should be guaranteed by steel (stainless austenitic steel X2CrNiMON22-5-3) substrates. With respect to low porosity levels, Al_2O_3 coatings were produced by high velocity oxygen-fuel (HVOF) spraying. In addition, detonation gun (D-Gun) spraying was employed as a bench mark.

Table 1 Powders and cold spray parameters

Experiment	Material	Size distribution, µm	p_{gas}, MPa	T_{gas}, °C
Wipe-test	Cu	−22 + 5	3	500
Coating	Cu	−35 + 15	3	600
Interlayer	Al	−45 + 20	3.5	420

Before cold spraying, the samples with ceramic layers were cleaned in an ultrasonic bath filled with ethanol, and dried carefully. Cold spray experiments were performed using the prototype of the Kinetics 8000 cold-spraying system. Copper was sprayed with a standard de-Laval-type nozzle (WC-Co, type 24, HSU/CGT, Germany). To avoid nozzle plugging, cold spraying of aluminum bond coats was performed with a PBI nozzle (type 33, HSU/CGT, Germany). More details on spraying parameters are specified in Table. 1, in which p_{gas} indicates the pressure and T_{gas} the temperature of the process gas nitrogen.

To examine the deformation behavior of a single copper particle when it impacts the ceramic surface, so-called wipe-tests were performed. In these tests, a polished substrate was moved rapidly through the spray jet. Afterward, the sample was investigated using Scanning Electron Microscopy (SEM, Phillips PW4860/00 XL40). The wipe-tests were performed using typical cold gas-spraying conditions and a fine Cu particle size fraction to examine the effects of damage caused even by small particles impacting on the brittle ceramic surface.

For heating the coated substrates during the spraying process, they were affixed to an electrical heating plate, and the temperature of the substrate was measured with a thermocouple element fixed in a drilled hole at one side of the sample, in addition to the control by the internal thermocouple.

For analyses of the microstructures and the bonding qualities of the cold-sprayed layers, cross sections were cut out of the samples and metallographically prepared by several grinding and polishing steps. The polished cross sections were investigated by optical microscopy (OM).

Electrical conductivity measurements were performed according to ASTM E1004 using an eddy-current measurement system (Sigmascope SMP 10-HF with sensor ES40HF at 1250 kHz, by Fischer, Germany), which was used for the layers in the as-sprayed state as well as for annealed layers. Annealing of the samples was performed for 1 h in an evacuated oven at a temperature of 500 °C and a pressure of 3×10^{-6} MPa.

3. Results

3.1 Impact Morphologies

Impact morphologies can supply vital information on bonding features in cold spraying (Ref 4, 8). In particular, such information is important for the expected high deformation of a ductile particle impacting on a hard and brittle substrate. For this, single impacts of copper particles as obtained by wipe-tests were investigated by SEM (Fig. 3, 4).

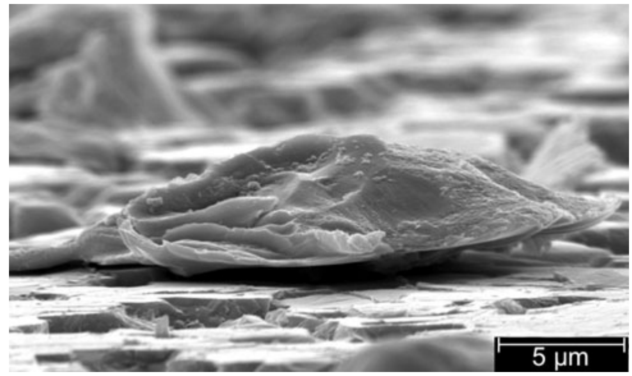

Fig. 3 Impact morphology of a single copper particle on a polished HVOF-sprayed Al_2O_3 layer under perspective view. The particle is highly flattened but not well bonded (SEM micrograph)

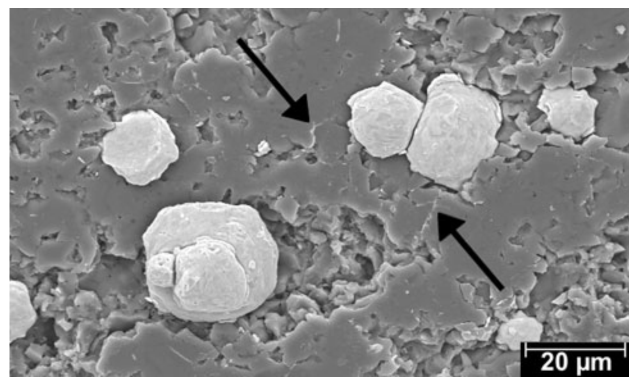

Fig. 4 Top view of impact morphologies of cold-sprayed single copper particles on a polished HVOF-sprayed Al_2O_3-layer. Arrows indicate cracks in the ceramic surface (SEM micrograph)

The perspective view of a single Cu particle in Fig. 3 shows pronounced flattening of the particle upon impact. Kinetic energy is fully converted into plastic deformation of the ductile particle, whereas the ceramic substrate seems not to be deformed at all. The break-outs of ceramic material from the surface (see in the front of Fig. 3) are probably caused by the former polishing procedure and are not caused by impacting particles. Another important observation concerns the shape of the characteristic jets at the margin of the particle. These jets are due to the viscous flow of material, which is caused by shear instabilities. In previous studies, the occurrence of shear instabilities at the edge of the particle was identified as the necessary precondition for bonding (Ref 2). In the present case, a zone of shear instabilities is in fact present, so that impact conditions and accordingly spraying parameters are chosen correctly. However, as compared to impacts on deformable substrates, the zone is much thinner and not in contact to the substrate surface. Thus, the particle is not sufficiently well bonded to the ceramic layer. Other important features concern possible failure of the ceramic layer under the high mechanical loads under the impact of copper particles. For distinguishing internal defects of the ceramic layer and fracture by impacting copper particles, the wipe-tests samples were analyzed in top-view, as shown in Fig. 4.

The SEM micrograph in Fig. 4 shows several copper particles (bright) on the surface of the polished Al_2O_3 layer (grey). Arrows indicate cracks in the ceramic surface, which are assumed to be induced by the impacting particles. Cracks of the indicated shape are commonly observed in the vicinity of the particles, and also in regions where remains of re-bounded copper particles are observed. The polished surface itself rarely shows such cracks, although it can not be excluded that the polishing process could induce cracks as well. To avoid damaging of the ceramic surface by solid impacts, either cold spray parameters have to be tuned to less harsh conditions or the toughness of the ceramic coating has to be improved. For ensuring good coating qualities, probably a compromise of both must be considered.

3.2 Coating Adhesion

According to the conclusions regarding the wipe-tests, spraying of a closed copper layer on a ceramic coating appears to be very difficult and requires careful studies and investigations. Figure 5 shows an example of a cold-sprayed copper coating on an Al_2O_3 coating that was processed by D-gun spraying. D-gun-sprayed coatings generally have higher deposition temperatures than HVOF coatings, resulting in higher densities and mechanical properties closer to bulk alumina than those obtained for HVOF coatings. Therefore, these coatings can serve as bench marks to explore the opportunities of deposition on thermally sprayed ceramics. For cold spraying, the process gas temperature was increased to 600 °C to account for the coarser feedstock powder. The cold-sprayed copper layer was well built up with very low porosity. However, it was not adhering well to the ceramic surface. Already, during preparation of cross sections, the copper coating got detached from the underlying Al_2O_3 layer.

This poses the main challenge, which has to be faced in cold spraying of metals on ceramic surfaces. Regarding cold spraying of Cu on HVOF-Al_2O_3, more restrictions of cold spray parameters are to be expected, compromising electrical quality of the Cu coatings. Thus, activation of the ceramic surface layers seems to be inevitable. In this study, this was done in two different ways: First, an aluminum interlayer was used as a bond coat. Second, the substrate was heated during the cold-spray process.

Figure 6 shows the cross section of a sandwich structure, in which a cold-sprayed aluminum interlayer serves as bond coat between the D-gun-sprayed Al_2O_3-ceramics and the cold-sprayed copper coating. All interfaces (Cu/Al and Al/Al_2O_3) adhere well. Since cold spraying of copper was performed with the same parameter settings as the detached coating, as shown in Fig. 5, the micrograph reveals the role of the aluminum interlayer as bonding agent between copper and Al_2O_3. Both cold-sprayed coatings, of copper and aluminum, are dense, and at the interface, single copper particles get immersed into the softer aluminum. Regarding the interface of Al and Cu, bonding can as well be explained by the model of shear instabilities.

The use of an interlayer as bondcoat involves an additional step in the production process. Thus, an alternative way to achieve the activation of the ceramic surface was investigated. The experiments indicated that such activation can be obtained by heating the substrate during the cold-spray process. Figure 7 shows cold-sprayed copper coatings that were processed on heated substrates. Temperatures were adjusted by a heating plate on the backside of the ceramic/steel substrate, and the substrate temperature was controlled by a thermocouple. At a low temperature of 120 °C, copper particles adhere weakly: For the most part, single passes of sprayed copper are removed by the gas stream of the next spraying line, so that parts of the ceramic surface are exposed again, and no closed Cu coating can build up (Fig. 7a). Increasing the temperatures to 230 °C leads to the formation of a copper coating which is peeled off the surface by the gas stream as well. In this case, flakes of copper with remains of Al_2O_3 at the backside (not shown in Fig. 7b) are detached,

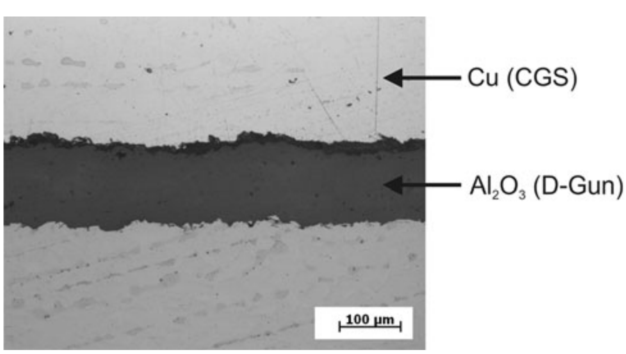

Fig. 5 Polished cross section of a cold-sprayed copper coating (top) on a D-gun-sprayed Al_2O_3 layer (middle). The copper coating is build up well, but already detached from the ceramic surface (OM micrograph)

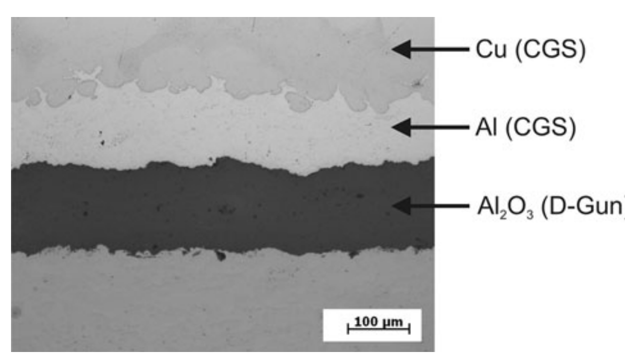

Fig. 6 Cross section with polish of a copper coating on D-gun-sprayed Al_2O_3-layer, with aluminum as bond coat. Aluminum and copper coatings are well build up and bonded (OM micrograph)

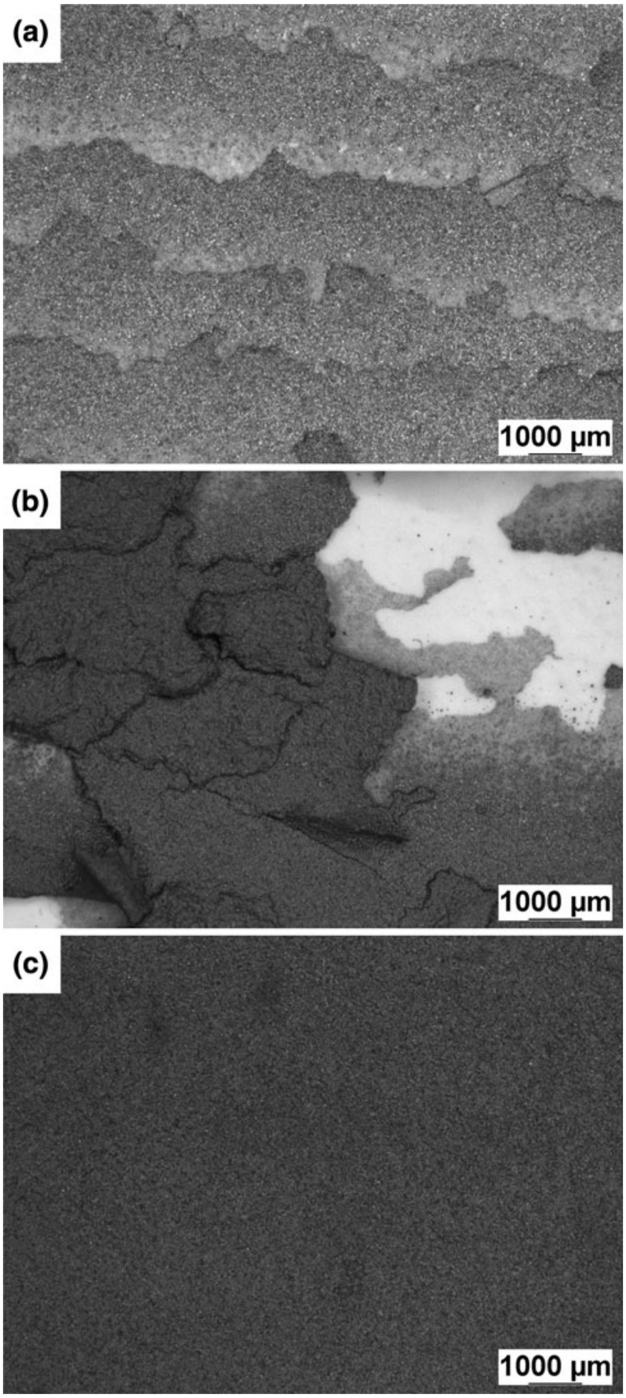

Fig. 7 Top views of cold-sprayed copper layers on HVOF-sprayed Al_2O_3 coatings, which were heated to different substrate temperatures of (a) 120 °C, (b) 230 °C, (c) 280 °C during the cold spray process (OM photograph)

leaving a clean, white ceramic surface. Heating the substrate to 280 °C, Fig. 7(c) and corresponding cross section in Fig. 8 results in a well-adhering copper coating, which slightly oxidizes at the surface as soon as the nitrogen gas stream has passed.

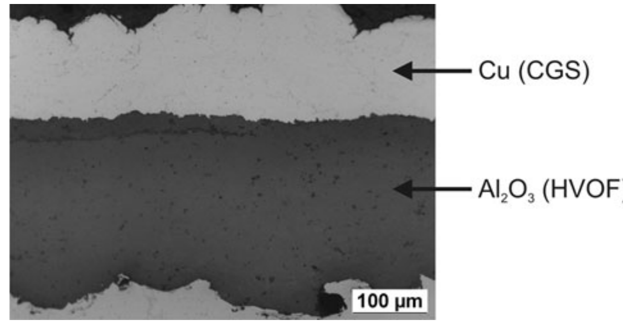

Fig. 8 Cross section of Fig. 7(c). Cold-sprayed copper coating on a HVOF-sprayed Al_2O_3 layer, which was heated to a substrate temperature of 280 °C during the cold spray process (OM micrograph)

For cold spraying on the substrate that was heated to 280 °C, the cross section in Fig. 8 shows a dense copper layer, which is well bonded to the ceramic substrate. Inside of the HVOF-sprayed Al_2O_3 coating, cracks arise parallel to the interface to the cold spray copper layer. These cracks are supposed to arise because of additional internal stresses inside of the ceramic layer generated by the impacts and thermal stresses induced by cold spraying. Thermal stresses are likely to occur because of the different thermal expansion coefficients of copper and aluminum oxide ($\alpha_{L,Cu} = 16.4$ µm/m °C, $\alpha_{L,Al_2O_3} = 5.5$ µm/m °C, both for room temperature; Ref 29). The fact that stresses cause fracture inside of the ceramic coating and not in the interface indicates a good adhesion between copper and Al_2O_3 for the present case. A good adhesion by cold spraying on heated ceramic is further substantiated by the observation in Fig. 7(b), in which copper layers get detached along with remains of the ceramic surface.

3.3 Electrical Conductivity

Mechanical integrity of the bilayer coating and functional properties will determine, which of the presented strategies for surface activation is more useful for practical applications. On the one hand, mechanical properties—particularly of the ceramic layer—will determine the long-time stability of the final component. On the other hand, the electrical conductivity of the copper coating will also be of great importance, since it reduces unnecessary heat, and thus energy losses.

Measurements of the in-plane electrical conductivity of a copper layer sprayed onto a heated substrate (as in Fig. 7c) gave values of more than 90% of the international annealed copper standard (IACS = 58 MS/m; Ref 30), for example, the electrical conductivity of the copper layer in Fig. 7(c) was 56.8 ± 0.9 MS/m, which was actually 98% IACS. Compared to this, the electrical conductivity of copper coatings sprayed on ceramic substrates at room temperature using aluminum as bond coat, see Fig. 6, was around 75% IACS in the as-sprayed state (43.0 ± 0.1 MS/m in the case of Fig. 6). This value is comparable to the electrical conductivities of cold-sprayed copper coatings in the as-sprayed state as measured in former studies (Ref 1, 31-33), which reported values between 61% IACS (Ref 32) and 82% IACS (Ref 33). The electrical conductivity could be improved significantly by a heat treatment for 1 h at 500 °C in an evacuated oven at pressures of 3×10^{-6} MPa. After this treatment, electrical conductivities of about 90% IACS were measured (52.6 ± 0.6 MS/m, after grinding preparation of the surface to remove oxide layers). This value corresponds well with results from literature (Ref 1, 32, 33), which range between 90% IACS (Ref 32) and 96% IACS (Ref 33), depending on the annealing procedure.

4. Discussion

The results of this study clearly showed the importance of surface activation for improving adhesion in cold gas spraying of metals on ceramics. The reasons will be discussed in the following.

Up to now, it is not fully understood why an aluminum coating can be prepared on the ceramic substrate, while a copper coating cannot be done so. One possible explanation is that impacts of aluminum particles impose less stress onto the ceramic layer due to their smaller density and momentum. Therefore, ceramic splats do not flake off and an aluminum coating could build up, which in turn works as a damping interlayer for later impacting copper particles. Assuming an average particle diameter of 25 μm for copper and of 32 μm for aluminum and considering the spraying conditions of Fig. 6, numerical calculations give comparable particle impact velocities of 577 and 599 m/s, respectively. This means that the copper momentum is about 1.5 times greater than that of the aluminum ($\rho_{Cu} = 8920$ kg/m^3, $\rho_{Al} = 2700$ kg/m^3; Ref 29). Thus, if the mechanical properties are inadequate to withstand the particle impact, brittle fracture of a thermal-sprayed alumina coating could occur. In contrast, bulk alumina substrates have higher fracture strength, so a cold-sprayed aluminum coating have been successfully built up, although the coating adhesion was very weak. This may be attributed to unfavourable distributions of residual stresses in the aluminum coating, which are released during detaching from the bulk alumina substrate.

Another possible explanation for the better adhesion of Al on the ceramic coating relates to corresponding chemical interactions between Al_2O_3 and Al or Cu. The thermodynamic stability of aluminum oxide is much higher than that of copper oxide: $\Delta H_f = -335$ kJ/g-atom-O in Al_2O_3 versus $\Delta H_f = -78$ kJ/g-atom-O in CuO (Ref 34). Thus, Al may be expected to bond chemically more easily to Al_2O_3 than Cu, even during the short heat pulse from shear instabilities upon impact.

Substrate heating was used as an alternative surface activation method. Heating of the ceramic substrate to temperatures of about 280 °C noticeably increased the bond strength of copper coatings. In thermal spraying, increased substrate temperatures are known to improve bond strengths as well (Ref 23-25). Weak bond strength is attributed to adsorbates and condensates formed on the surfaces from the ambient air that could significantly hinder the bonding process (Ref 12-22). It can be assumed that heating of the ceramic layer may reduce the amount of water at the surface, allowing intimate contact between copper and the Al_2O_3 ceramic and therefore supporting adhesion in thermal spraying as well as in cold spraying. Moreover, the results also indicate that crack propagation in the ceramic layer seems to be less critical at higher temperatures.

Apart from surface activation, the surface design of the ceramic coating could have a significant effect on successful adhesion of cold-sprayed particles as well. Figure 4 suggests that particles are bonded, or at least they remain on the substrate, in rough surface areas. Therefore, a successful coating build-up could be assisted by a certain surface roughness of the ceramic substrates, compare Fig. 3 and 4. This observation leads to two different interpretations regarding the general bonding mechanism between metal and ceramic in cold gas spraying: On the one hand, mechanical anchoring between copper particles and the ceramic surface might be identified as the main adhesion mechanism. Nevertheless, such mechanical interlocking without chemical bonding is expected to lead to a relatively weak coating adhesion. On the other hand, the model of shear instabilities could also be regarded as an explanation for bonding, as observed in the wipe-test: As shown in Fig. 3, shear instabilities arise at the edges of the impacting copper particles. However, these zones are not in contact with the substrate, and therefore the particle is not well bonded on smooth ceramic surfaces. In contrast to smooth surfaces, zones of shear instabilities being in contact with the substrate could be increased for rough surfaces. Increased contact areas can in turn lead to higher adhesion strengths of coatings. This adhesion is assumed to be because of a chemical bond forming in the zones of shear instabilities, in which the temperature at the particle-substrate interface is dramatically increased during the short duration of impact (Ref 4).

From the wipe-test results, it cannot be deduced, which of the different adhesion mechanisms, namely, mechanical anchoring or chemical bonding in zones of shear instabilities, is a realistic model to describe the bonding between cold-sprayed copper and ceramic substrate. However, in section 3.2, it is described that the coating adhesion can be improved by increasing substrate temperature during spraying. In the case of mechanical anchoring, the substrate temperature should not affect the coating adhesion. In contrast, substrate temperature is expected to have an effect on chemical bonding because of removal of adsorbates or condensates, which could hinder the successful formation of a chemical bond between the particle and substrate surface, as observed in thermal spraying (Ref 12-22). Moreover, higher substrate temperatures can enhance interdiffusion or chemical reactions. Summarizing the observations of the wipe-tests combined with the results in section 3.2, the model of chemical bonding in zones of shear instabilities is favored to explain the adhesion of cold-sprayed metals on ceramics.

To demonstrate the potential of cold-sprayed copper coatings for electronic applications, electrical conductivity measurements were performed. In summary, tempering of copper coatings sprayed on ceramic substrates using aluminum as bond coat results in electrical conductivities, which are close to conductivities of cold-sprayed copper on heated ceramic substrates. Therefore, both surface activation alternatives are comparable with respect to their electrical properties.

Regarding adhesion and coating build-up, the influence of residual stresses in both cold gas-sprayed metal coatings as well as in thermal-sprayed ceramic layers requires further investigations.

5. Conclusions

This study demonstrates that metallic coatings with high electrical conductivity can be successfully deposited onto insulating thermal spray ceramic coatings by cold gas spraying. Good adhesion is achieved via an activation of the ceramic surface. As in thermal spraying, heating of (ceramic) substrates is beneficial to remove adsorbed water to support bonding. Moreover, the use of chemical activation by interlayers can improve coating build-up, despite the short time scales during particle impact in cold gas spraying. By both alternatives, dense copper layers can be produced. The electrical conductivity reaches 98% IACS in the as-sprayed condition on heated substrates, or 90% of the IACS value after spraying on cold substrates with aluminum bondcoat and additional heat treatment, thus meeting the requirements for electronic applications.

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