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Consolidation of Al₂O₃/Al Nanocomposite Powder by Cold Spray

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While the improvement in mechanical properties of nanocomposites makes them attractive materials for structural applications, their processing still present significant challenges. In this paper, cold spray was used to consolidate Al₂O₃/Al nanocomposite powders obtained from mechanical milling. The microstructure and nanohardness of the feedstock powders as well as of the resulting coatings were analysed. The results show that the large increase in hardness of the Al powder after mechanical milling is preserved after cold spraying. Good quality coating with low porosity is obtained from milled Al. However, the addition of Al₂O₃ to the Al powder during milling decreases the powder nanohardness. This lower hardness is attributed to non-optimised milling parameters for proper Al₂O₃ embedding and dispersion in Al and results in a lower coating hardness compared with the milled Al coating. The coating produced from the milled Al₂O₃/Al mixture also shows lower particle cohesion and higher amount of porosity. The overall results are promising and it is believed that an optimization of Al milling with Al₂O₃ will allow production of sound coatings with improved hardness.

1 Introduction

Due to their lightweight and high specific strength, aluminum matrix composites are attractive structural materials for various domains such as automotive and aerospace applications. Recently, several authors have measured an increase in strength in aluminum or magnesium composites when the reinforcement size goes from microscale to nanoscale and it has also been reported that this strengthening is combined to an increase in ductility [1-5]. On the other hand, the processing of composites with nanoreinforcement is not straight forward. In powder metallurgy, high energy mechanical milling was found to eliminate nanoparticle agglomeration and clustering issues for several composites [2,6,7]. In addition to homogeneous reinforcement dispersion, mechanical milling induces a significant grain size reduction, also beneficial to strength properties. However, the resulting milled powders were found to be difficult to consolidate. The compacts obtained by traditional consolidation methods such as hot pressing or extrusion display high porosity and poor particles bonding. Furthermore, the methods involving application of heat can lead to the loss of the nanograin structure as well as unwanted reactions [8, 9].

Cold spray technique consists in a coating production with high-velocity particles at temperature significantly lower than the melting temperature of deposited materials. Due to the low temperatures involved in the process, cold spray can consolidate mechanically milled composite powder while preserving their nano grain size [10-13]. For grains with size above the lower limit achievable by deformation, cold spray can even further refine the grains [13]. Cold spray has also been used successfully to produce a few nanocomposites: dense coatings of TiB₂-Cu [14], TiN-Al [15] and Al₂O₃-Cu [16] were obtained. In all cases, a significant increase in hardness was obtained when compared with the equivalent unreinforced and unmilled coating.

The objective of this study is to assess the potential of cold spray to consolidate alumina/aluminum (Al₂O₃/Al) nanocomposite powder produced by mechanical milling. The spraying parameters have first been adjusted for milled powders. The microstructure and properties of the resulting coatings were then studied.

2 Experimental Procedure

Al powder of -325 mesh, 99.5% purity, from Alfa Aesar (Ward Hill, USA) and spherical Al₂O₃ of nominal size of 4 nm bought from Aldrich (St-Louis, USA) were used to produce the composite powders. **Fig. 1** shows the initial materials.

Fig. 1. SEM micrographs showing: (a) Al₂O₃ powder and (b) initial Al powder.

Two mechanically milled mixtures were produced, one from Al only and another one composed of 5 vol% Al₂O₃/Al. Milling was performed in a Retsch (Haan, Germany) planetary mill PM200 with a ball-to-powder ratio of 7.5:1, a speed of 450 rpm and a milling time of 15 hours under argon atmosphere. For ductile materials such as aluminum, a process control agent, stearic acid in this study, is added during milling to prevent excessive welding. The appropriate powder size distribution for cold spray technique was adjusted by varying the amount of stearic acid between 1.5 and 6 wt%. The objective was to obtain a unimodal size distribution with an average as close as possible to 30 microns and a low amount of particles exceeding 60 microns [17]. At the end, 4 wt% stearic acid was selected, leading to an average

size of around 35 μm and a particle morphology between equiaxed and flaky (**Fig. 2**). **Fig.3** shows the size distribution of the milled powders. The milled powders were heat treated (HT) at 450°C for 15 minutes before cold spray.

Fig. 2. SEM micrograph showing milled $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ powder.

Fig. 3. Particle size distribution of the milled powders.

The milled $\text{Al}+\text{HT}$ and milled $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ powders as well as the initial Al powder were cold sprayed on grit blasted mild steel substrates to produce three coatings. The final cold spray parameters are as followed: Inovati (Santa Barbara, USA) cold spray system using Kinetic Metallization technology (a cold spray variant where a convergent nozzle is used to accelerate the process gas to Mach 1 [18]), inlet helium temperature and pressure of 500°C (315°C for initial Al) and 0.78 MPa, standoff distance of 2 cm, transverse speed of 10 mm/s (50 mm/s for initial Al), powder mass flow rate of about 5 g/min. The selection of those parameters is further discussed in the result section.

Morphology, size and distribution of the Al_2O_3 and Al powders were studied using FE-SEM Hitachi S-4700 (Tokyo, Japan) and the particle size analyser Horiba LA-920 (Tokyo, Japan). The cold spray coating microstructures in cross-section were investigated with a SEM Jeol JM-6100 (Tokyo, Japan) after standard metallographic preparation. Phase change and precipitation were studied with the Bruker D8 Discover X-Ray diffractometer (Madison, USA).

Nanohardness of powders and coating was evaluated using the Nano G200 from MTS/Nano Instruments (Oak Ridge, USA) with a Berkovich tip. 15 to 20 indents were conserved after filtration. Peak forces of 1 or 5 mN were selected with a hold time of 10 s. Nanohardness was calculated from the maximum load divided by the projected contact area. The reduced elastic modulus was calculated from the stiffness following the Oliver-Pharr data-analysis procedure [21]. The reduced elastic modulus was then converted to the Young modulus from [19]:

$$\frac{1}{E_r} = \frac{(1-\gamma^2)}{E} + \frac{(1-\gamma_i^2)}{E_i}$$

where E and γ are Young modulus and Poisson ratio for the specimen, the latter being estimated as 0.33 [20]. E_i and γ_i are the same parameters for the diamond indenter, taken to be 1141 GPa and 0.07 respectively from [19].

3 Results

3.1 Effects of Milling and Heat Treatment on Powders

Fig. 4 shows the nanohardness obtained from the powders at various process stages as well as from the coatings. In order to minimize the effect of epoxy during indentation of the polished cross-section of mounted powders, a very small load had to be used. This has somewhat increased scatter in the measured values. It has also introduced a bias toward higher hardness values. This trend, referred as indentation size effect, has been observed elsewhere [21,22]. It is worth noting that the explanation for this phenomenon is still under debate. However, as a constant load was used for all samples, the results can be compared one with each other.

Fig. 4. Powder (P) and Coating (C) Nanohardness at 1 mN load

From **Fig. 4**, it is seen that milling increases the Al powder hardness. Deformation occurring during milling leads to cold working and grain refinement [23]. Also, various contaminants coming from the addition of the process control agent, wearing of the balls and vial, the initial oxide layers at the surface of the Al particles or the extra oxidation occurring during the milling process, are embedded in the Al powder where they can form either a solid solution or dispersoids [24]. All those phenomena contribute to the hardening observed after milling.

In the same Figure, it can also be noticed that the milled Al powder nanohardness is increased after heat treatment. XRD patterns of the milled Al powders has shown Al peak shift of 0.1° toward lower angles following heat treatment (**Fig. 5**). This shift is associated to a decrease in Al lattice parameter, probably from a lower amount of atoms in solid solution. Upon heating, the impurity atoms in solid solution possibly precipitated out. It is believed that this precipitation is at the origin of the powder hardening after heat treatment. On the opposite, no sharpening of the peaks, indicating a possible annealing effect, was observed. It is supposed that the heat input during cold spray could have a similar effect on milled powders. This could lead to unusual trends such as a lower deposition efficiency with the use of a higher gas temperature. While a heat treatment was applied to all milled powders of this study to ease comparison between the properties of the powder feedstock and the coatings, an in-depth study of the effect of heat input during cold spraying could allow further understanding and optimization of the process.

Fig. 5. XRD patterns of milled Al powder before and after HT.

If the hardness of the milled Al+HT powder and the milled $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ powder are compared, it can be seen that the former powder is harder. This was unexpected; Al_2O_3 was added with the purpose of reinforcing the Al. Previous work with the same initial constituents but different milling conditions indeed led to an increase in hardness with Al_2O_3 addition [9]. It is believed that the modified milling parameters used in this study to accommodate cold spray

requirements in term of particle size and production yield did not allow optimised embedding and dispersion of Al_2O_3 in Al (typically obtained at milling “steady state”). **Fig. 6** shows the polished cross-section of milled $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ powder. Stack of poorly welded particles are clearly seen, together with porosity and cracks. This lack of cohesion between lamellas could also lower hardness.

Fig. 6. Polished cross-section of milled $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ powder.

3.2 Spraying Parameters for Milled Powders

In order to cold spray a powder, the powder must first be fed to the nozzle. The flaky morphology of the milled particles reduces powder flow and has made powder feeding impossible with the CGT Kinetic 3000 cold spray system. The effect of powder morphology on powder feeding has been studied by H. Gruner et al. [25]. This study showed that powders with an angular morphology have large surface to volume ratios which would make the powder feeding during thermal spray, including cold spray, difficult (bad powder flow and tendency to build up bridges, blockages or pulsative powder flow). These problems were not observed with powders having a spherical morphology.

In order to favor powder flow, Inovati cold spray system was selected. The feeding system contains a brush that deagglomerates the powder and pushes it through a screw. By using the highest mesh screen (20 mesh) and the hopper maximum speed (100%), a feeding rate of 5 g/min was achieved for milled Al+HT and 4.6 g/min for milled $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ mixture. In comparison, the initial Al powder, with an equiaxed shape, only necessitates 15% of the hopper speed for a feeding rate of 4.6 g/min.

The second challenge encountered during cold spraying of milled powders is their higher hardness that reduces their ability to deform upon impact with the substrate [26]. In this study, typical conditions led to no deposition. To favor deposition, the gas temperature was raised to the highest temperature allowable, 500°C . By raising the gas temperature, the particle velocity is increased and the particle temperature also rises to allow more deformation upon impact. The transverse speed of the robot was also reduced to 10 mm/s to heat up the substrate.

The efficiency of deposition stayed very low, with a value of 15% for milled Al+HT and 7% for the milled $\text{Al}_2\text{O}_3/\text{Al+HT}$. In comparison, the deposition efficiency of initial Al was of 43% with a lower gas temperature of 315°C and a higher transverse speed of 50 mm/s.

It's worth noting that even if the milled $\text{Al}_2\text{O}_3/\text{Al+HT}$ powder had a lower hardness than the milled Al+HT powder, it got a much lower deposition efficiency. The Al_2O_3 particles could reduce the ductility of the powder locally, impeding proper deformation of the splat. Al_2O_3 at the surface of the Al powder could also impede physico-chemical reactions contributing to particle cohesion, similarly to what was observed elsewhere for surface impurities [27].

3.3 Coating Microstructures

The microstructures of the three cold sprayed coatings are shown in **Fig. 7**. The coating produced from the initial Al powder is dense: minimal porosity is visible. As opposed to the milled Al+HT and milled $\text{Al}_2\text{O}_3/\text{Al+HT}$, the coating also remained strongly attached to the substrate through the cutting and mounting process involved in metallographic preparation, indicating a better adhesive strength.

Fig. 7. Polished coating cross-sections at two magnifications: (a) and (b) Initial Al coating, (c) and (d) Milled Al+HT coating, (e) and (f) Milled $\text{Al}_2\text{O}_3/\text{Al+HT}$ coating.

Milled Al+HT coating display smaller but more numerous pores than the initial Al coating. While good quality coatings can be obtained with nanostructured feedstock, the porosity in the latter is

always higher than in conventional coatings. The difference in porosity is explained by the higher hardness of the feedstock powder. Plastic deformation is limited and generalized adiabatic shear instability is not experienced by the milled powder, resulting in a porous coating [12]. **Fig. 7 (b)** shows one of the few interlamellar cracks observed between transverse passes in the milled Al coating. Those cracks indicate a lack of bonding between adjacent deposited layers. By decreasing the robot transverse speed for milled powders, the amount of powder deposited for each transverse pass was increased. Because of the formation of the coating, the impingement angle for the particles of the subsequent pass was increased, leading to a lower normal velocity component. It is believed that the cracks result from the reduction in the extent of deformation due to the decreased normal velocity component.

The milled $\text{Al}_2\text{O}_3/\text{Al+HT}$ coating is half the thickness of the milled Al coating due to the poor deposition efficiency. The coating is very porous, cracked and brittle, showing poor particle cohesion. A fractured cross-section of the coating was also observed under FE-SEM. **Fig. 8** shows that the Al_2O_3 particles were indeed deposited in the coating. The embedding of the reinforcement in the matrix powder during mechanical milling should minimise loss of reinforcement during spraying. Al splats are clearly visible. The bonding between those splats seems to be weak. This lack of bonding could be due to the Al_2O_3 particles present at the surface of the Al splats that would impede physico-chemical interactions at the origin of cohesion. The numerous Al_2O_3 particles present at the surface of the splats rather than embedded within the Al could be due to insufficient milling.

Fig. 8. Fracture surface of the Milled $\text{Al}_2\text{O}_3/\text{Al+HT}$ coating.

A thin film produced out of the cold sprayed milled Al coating was observed with a Philips CM200 TEM. **Fig. 9** shows a region where elongated and curved grains (highlight by arrows) were observed in the milled Al+HT coating. Those elongated grains are similar to those observed in adiabatic shear bands produced from high strain rate deformation [28]. The non-uniform coating microstructure composed of coarse, fine and elongated grains support the adiabatic shear instability mechanism proposed for

deformation occurring during cold spray. While the coarse grains would correspond to particle region where very little deformation has occurred, as in the middle of the particle, the fine grains (size of about 90 nm) would correspond to areas of intensive plastic deformation where recrystallisation has occurred, i.e. the regions of localized shearing at particle interfaces [29]. The elongated and deformed grains would be intermediate regions where deformation has occurred, but not to an extent sufficient to trigger recrystallisation.

Fig. 8. TEM micrograph of the milled Al coating.

3.4 Coating Hardness

Coating nanohardness can be compared with powder nanohardness in **Fig. 4**. For each powder type, an increase in hardness is observed after cold spraying. Part of this increase is probably due to the work hardening occurring during powder impact on the substrate. However, other factors might be involved, such as closing of cracks and porosity presented in the milled powders upon impact deformation or removal of the epoxy compliance effect in the bigger coating samples.

In order to improve accuracy, the coating nanohardness were also tested at higher load and with the Dynamic Contact Module (DCM) head of the nanoindenter, with higher resolution. The results are displayed in **Table 1**. It can be seen that the elastic modulus is the same for the three coatings. This is as expected because the coating compositions are similar. It also means that the load used is small enough to avoid the effect of coating porosity. If this was not the case, the elastic modulus of the $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ coating would be lower due to the higher porosity level. Furthermore, no crack or porosity was observed at the indents. The elastic modulus values obtained are slightly higher than Al Young modulus measured through tensile test (70 GPa [30]) and similar to the values obtained by nanoindentation from another study (80 GPa [31]).

Table 1. Coating Nanohardness and Elastic Modulus at 5 mN load

Coating	Nanohardness 5 mN (GPa)	Elastic Modulus (GPa)
Initial Al	0.96±0.09	83±4
Milled Al+HT	2.1±0.1	76±3
Milled Al_2O_3 +HT	1.3±0.3	82±4

Milling of the initial Al powder doubles the resulting Al coating nanohardness. The powder gain in hardness after milling is therefore preserved after cold spraying. The higher gas temperature used for the spraying of milled powders compared with the gas temperature used for the initial Al powder could also play a role. As a higher gas temperature results in higher particle velocity, the particles could deform more upon impact, increasing the extent of cold working.

The $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ coating displays a higher nanohardness than the initial Al coating, but a lower nanohardness than the milled Al coating. The nanohardness ranking of the powders is then reflected in the coating and it can be expected that $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ coating hardness could be much improved with feedstock of higher quality.

4 Conclusions

In this study, the potential of cold spray to consolidate $\text{Al}_2\text{O}_3/\text{Al}$ nanocomposite powder produced by mechanical milling was assessed. It was found that it is possible to cold spray milled powders once the feeding and deposition issues are tackled. The increase in hardness of Al powder due to milling is preserved after cold spraying. Good quality coatings with low porosity can be obtained.

However, the addition of Al_2O_3 to the milled powder further decreases deposition efficiency and leads to porous and brittle coatings, with lack of bonding between particles. $\text{Al}_2\text{O}_3/\text{Al}+\text{HT}$ mixture display improved hardness compared to initial Al powder. Yet, its hardness is lower than the milled Al powder hardness due to the non-optimized milling parameters. This tendency is preserved in coating hardness. This study shows that cold spray can consolidate $\text{Al}_2\text{O}_3/\text{Al}$ nanocomposite powder produced by mechanical milling. Nevertheless, it is believed that the powder feedstock needs to be improved and an upcoming optimisation of mechanical milling parameters should allow production of better quality nanocomposite coatings with improved hardness.

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