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Lasers and Thermal Spray

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Abstract. Basically, thermal spray and laser processing can be considered as half brothers since they show many common features due to the use of a (more or less) high-energy source for both. Their combination can therefore be very fruitful and prominent to achieve coatings, which results in their most recent and advanced applications. In the materials processing development story, the laser will thus have moved from cutting to coating. This keynote presentation focuses on the recently-developed coupling of laser processing to cold spray). In this dual process, a cold spray gun is combined to a laser head in a single device, e.g. on a robot. Series of coating experiments using various laser irradiation conditions, primarily pulse frequency, were carried out for Al-based and Ni-based alloys. Laser pre-treatment of the substrate just prior to cold spray, was shown to be beneficial for adhesion of cold-sprayed coatings. Adhesion improvement was exhibited and studied from LASATesting (LASAT for "LAser Shock Adhesion Test"). Incidentally, through LASAT also, the role of lasers in the development of thermally-sprayed coatings can be considered as major. Results are discussed in the light of a TEM (Transmission Electron Microscope) study of the coating-substrate interface with and without laser pre-treatment.

Introduction

Thermal spray is often considered as a basic and low-advanced process (Fig. 1). One may refer to the so-called "Schoop process" which still conventionally term a good deal of spray processes even though they have very little to do with this early and historical metallizing process [1]. Combining laser processing to thermal spray can give the latter an illustrious history through a better efficiency and a high–tech touch. On the reverse, this combination is also good for lasers to move from its former and major application of cutting (Fig. 2) to that of coating, in a "cut-to-coat" evolution. Laser means there high-power lasers to be used for treating materials, which exclude lasers as tools for diagnostics. Combining laser processing to thermal spray is the best for materials development despite they can be considered as false friends due to several/many potential overlapping areas for applications and due to many common features, as already discussed in a previous review article by the first author [2].

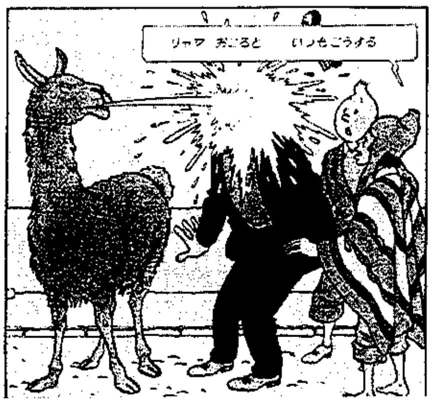


Fig.1. “P*llama* spray” as an illustration of spray processing, after Hergé 1949, [3].

Fig.2. An illustration of laser processing, after “Goldfinger” in the 007 series, 1953, Courtesy of UnitedArtists.

Combining laser processing to thermal spray results in a major improvement for thermal spray in 3 sub-areas, i.e. that of simulation of thermal and kinetic phenomena, that of post-treatment and that of pre-treatment. First, a laser can be used as a tool for *simulating* thermal deposition from another medium (e.g. an electric arc or a high-temperature droplet) onto a thermally-sprayed coating [4]. A laser can also be used for simulating shock phenomena involved in cold spray to lead to an original and recent approach to this process [5]. Second, a high-power laser can be used for healing and sealing thermally-sprayed coatings through densification in a *post-treatment* stage. This can be obtained using conventional remelting or, more originally using laser shock processing, namely “laser peening” [6]. Still in the, one may also call, post-treatment domain, a laser can be used for thermal testing and for mechanical testing [2]. The latter is more advanced and original, though the recent development of Laser Shock Adhesion Testing, namely LASAT. LASAT already showed a high capability to test the adhesion of thermally-sprayed coatings, including that of single splats [7]. Third, the coupling of a high-power laser with thermal spray dates back to a certain number of years, with the development of several variants [2] for *pre-treating* the substrate in particular prior to spraying. The most popular process in this family is known under the name of PROTAL®, i.e. “PROjection Thermique Assistée par Laser” (Laser-Assisted Thermal Spray), despite there is not yet industrial applications [2]. In the wake of PROTAL®, which formerly involved conventional thermal spray , i.e. plasma or flame spray, the innovative dual process to be discussed in this article is based on the coupling of cold gas dynamic process with laser processing [8,9]. This combination is expected to be all the greater the spray process is carried out under “cold” conditions as in cold spray.

Cold spray is commonly claimed to be the most innovative process in the range of thermal spray processes. However, its development is still somewhat curbed due to a rather bad control of materials and processing conditions to result in a satisfactory coating-substrate adhesion although a high adhesion can be obtained in some cases actually [10]. This control is particularly uneasy since major materials interaction phenomena required for suitable adhesion are local and limited to shallow layers. The creation of fresh surfaces for the materials at the contact, primarily the substrate, can therefore be assumed to promote adhesion as suggested by earlier studies [11].

Consequently, in the present work, laser-assisted cold spray was developed to clean (in its general meaning) the substrate to improve adhesion. Series of cold spray coating experiments were conducted with and without laser pre-treating just prior to spraying. Two coating systems were studied, i.e. Al and Ni-20Cr onto an Al-based alloy and a Ni-based alloy respectively, to show the influence of materials and processing conditions on coating-substrate adhesion. The latter could be studied from LASATesting in addition to thorough observation of the substrate surface and the coating-substrate interface, including SEM, TEM and quantitative image analysis. Basic materials surface and interface mechanisms could be elucidated as a function of laser parameters.

Materials and Processes

Powders and Substrates. Commercial [+17,-35μm] Alfa Asear Al and [+20,-53μm] 1616-09/PS Ni-20Cr Höganäs powders were cold sprayed onto conventional AISI 2017 Al-based and Inconel 718 (IN718) Ni-based substrates of 20x50x3mm^3 typically. Hardness measurements were carried out on both powders and substrates, which gave: 28±5 $HV_{0.01}$ (Al) and 192±15 $HV_{0.05}$ (Ni-20Cr) for the powders and 110±4 $HV_{0.5}$ (Al 2017) and 271±7 $HV_{0.5}$ (IN718) for the substrates respectively.

Coating Process. In the cold spray-laser dual system, the laser head was coupled with the spray gun to result on laser passing just prior to powder deposition (Figs. 3a and 3b). The laser head was that used in a PROTAL® device. Spray/laser passing was always the same (Fig. 3c) to keep the same heat transfer from inlet gas to the substrate in all the experiments. Two passes were used to build the coating, the first scan with the laser on, the second with the laser off.

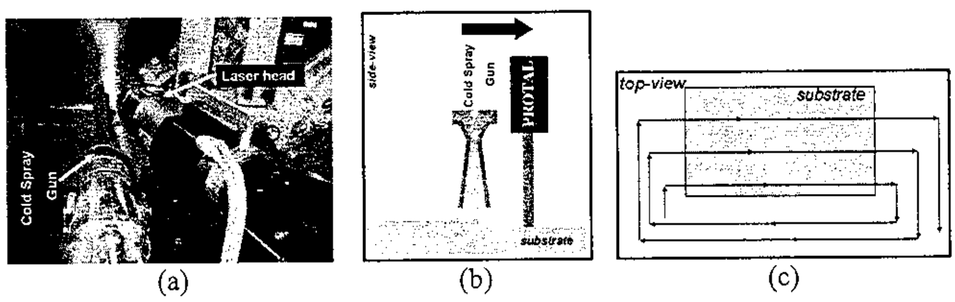


Fig.3. a) Experimental set-up, b) Schematic illustration of the coupled system, c) Gun scan.

Cold Spray. The best spraying parameters were selected from preliminary cold spray tests for both materials (Table 1). Spraying was performed with a PBI-33 polymeric nozzle (by CGT GmbH) and a conventional steel "MOC" nozzle respectively, using a KINETICS® 3000-M System by CGT GmbH.

Table 1: Spraying parameters.

Process gas	Gas pressure (MPa)	Gas temperature (°C)	Standoff distance (mm)	Gun traverse speed ($mm.s^{-1}$)
N_2	3.0	350	20	100

Preliminary tests included measurements of in-flight particles using a ColdSprayMeter® by Tecnar Automation Ltd., Québec), a few typical results of which are shown (Fig. 4) for IN718 only.

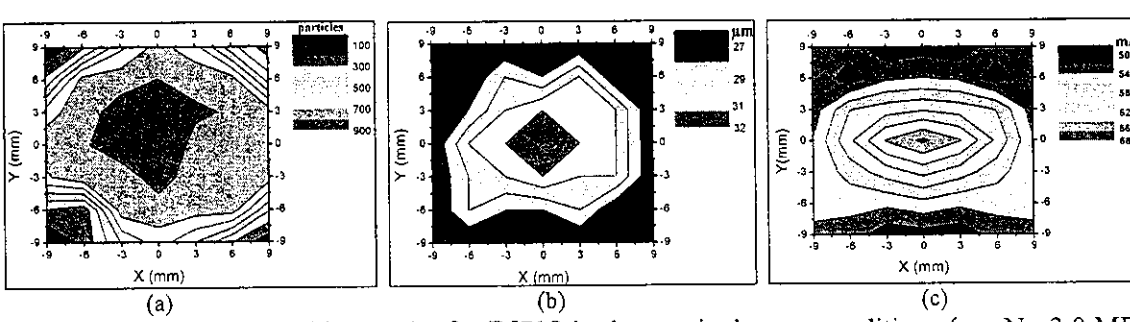


Fig. 4. Mappings of the cold spray jet for IN718 in the nominal spray conditions (gas N_2, 3.0 MPa, 600°C) , a) number of particles, b) particle diameter , c) in-flight velocity.

The coating material, i.e. Al and Ni-20Cr, was cold-sprayed onto as-received, grit-blasted (with 300μm angular alumina grit), mirror-polished, and laser-treated substrates.

Laser Pre-Treatment. The laser source was a Q-switched Nd:YAG laser (by QUANTEL/Les Ulis-France) which operated at 1.064 µm with an average output of 40 W for a pulse duration of 10 ns with a "top-hat" energy distribution. The laser spot was of 5x10 mm^2 and 3.5x7 mm^2 in dimensions for a laser energy of 1.0 $J.cm^{-2}$ and 2.2 $J.cm^{-2}$, respectively. Since the cold spray spot showed a diameter of 10 mm, the pattern of cold-sprayed particles was larger than or equal to that of the laser spot. (Fig. 5). Various pulse rates were tested, which corresponded to different overlap ratios (Fig. 5).

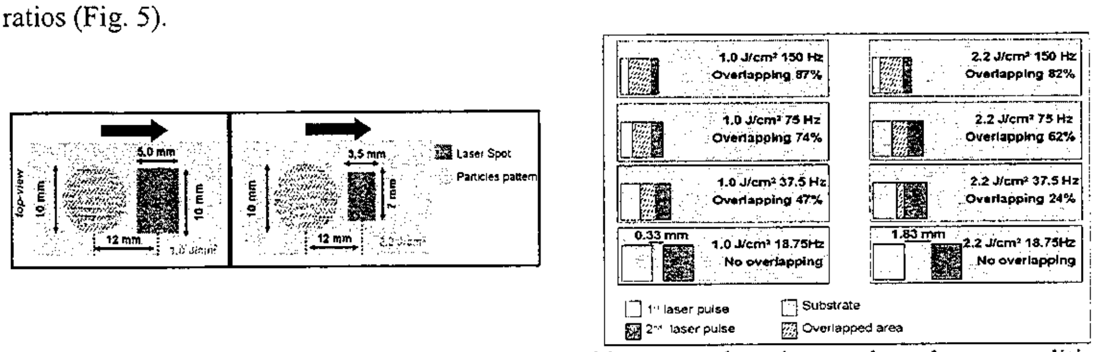


Fig. 5. Schematic illustrations of cold spray and laser spot locations vs laser beam conditions.

Laser pre-treatment was applied to the "as-received" state of Al 2017 and IN718 (Table 2).

Table 2. Laser treatment conditions prior to spraying onto Al2017 and Ni-20Cr substrates.

Substrate	Laser energy density (Jcm^{-2})	Pulse rate (Hz)	Overlapping of 2 pulses (%)
As-received	-	-	-
Polished	-	-	-
Grit-blasted	-	-	-
As-received	1.0	37.5	24
As-received	1.0	150	82
As-received	2.2	37.5	47
As-received	2.2	150	87

Characterization

Microstucture. In addition to conventional metallography, Scanning Electron Microscopy (SEM), and Quantitative Image Analysis (QIA) (with ImageJ® software), Transmission Electron Microscopy (TEM) was used to investigate into the coating-substrate interface. For this, thin foils of selected specimens were FIB-prepared. For this same selection, X-ray microtomography (XMT), was performed at the ESRF using beamline ID19. In this experiment a collimated X-ray beam at 17.6 keV penetrated the sample of 0.5×0.5×10mm^3 in size. Specific volume chemical etching was applied to the sample prior to XMT to exhibit microstructural features. The transmitted X-rays were collected using a 2D detector. The reconstruction of the 3D structure was obtained from the recording of several radiographs of the sample for different angular positions (1500 radiographs for 180°). Tomography was carried out with a resolution of 0.35 µm per voxel.

Coating-Substrate Adhesion. LAser Shock Adhesion Test (LASAT) was used to measure coating adhesion in the Al-Al2017 system. The principle is based on the irradiation of the rear surface of the substrate with a laser beam to generate a shock wave. This shock wave propagates and reflects in the material, which creates release waves, the crossing of which leads to tensile stresses at the interface. LASATestings were carried out using Nd:YAG laser, which delivered 40ns Gaussian pulses. The beam focused on a spot of 4mm in diameter at the rear surface of the substrate, in a wide range of laser power densities (1.7 to 4.3 $GW.cm^{-2}$). In addition, during the test, Doppler laser interferometry using a Velocity Interferometer System for Any Reflector (VISAR) was applied to the coating surface to measure its velocity as a function of time.

Results

Laser Surface Modification of the Substrate. Initial surface features, e.g. scratches for Al 2017 or structure cells in IN718, tend to vanish for both when submitted to the laser light. This was caused by superficial remelting which was all the more pronounced as the laser density and laser pulse rate increased. Craters could form from defects such as small inclusions, precipitates or cracks, which concentrated laser-deposited energy (Fig. 6).

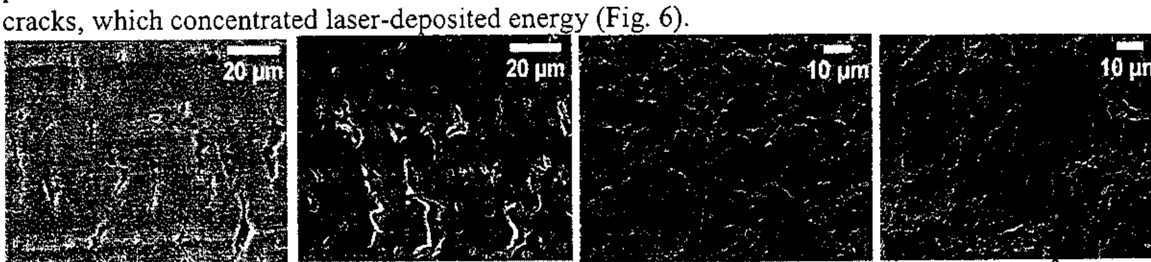


Fig. 6. SEM top views of a) and c) as-received and, b) and d) laser-processed (1.0 $J.cm^{-2}$, 150 Hz) Al 2017 (left) and IN 718 (right) substrates.

Coating Microstructure. A systematic study of coating thickness, coating porosity, coating composition, and coating microhardness, the main conclusion of which was given only. For detailed results, one should refer to [12]. This study used microscopes, quantitative image analysis and electron probe microanalysis. For Ni20Cr, the mean *coating thickness* was seen to be the same whatever substrate preparation, i.e. about 200 µm. In contrast, for Al, a significant increase, i.e. up to about 30% more, could be observed when increasing laser energy density, due to a more rapid build-up of the first coating layer due to better adhesion of the particles (Fig. 7).

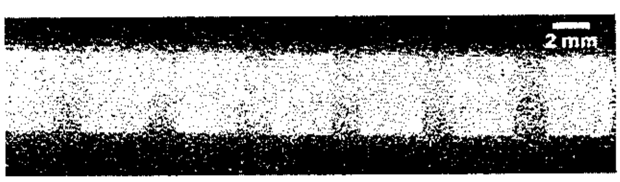


Fig. 7. SEM top view of the first pass of Al onto Al 2017 with a high power particle density in the laser-processed (bright) zones, 2.2 $J.cm^{-2}$, 37.5 Hz.

As for *porosity*, Al coatings were quasi-dense. Ni-20Cr coatings exhibited a lower porosity when using the laser, except for high energy density coupled with high pulse rate: i.e. around 2% against around 3.5 % (which was nearly that of coatings on untreated substrates). This can be attributed to some thermal effect up to a given level for the laser density, above which a counter effect (still to be elucidated) is supposed to occur. Whatever the conditions, coating density was always better that that obtained using conventional thermal (both flame and plasma) spray [13]. In addition, coating *composition* was for all free from oxides, which could explain a somewhat lower *microhardness* compared to that of conventional thermally-sprayed coatings. This effect was significant for Ni-20Cr coatings, as that of loading level. When Vickers-tested under 0.05 kg rather than under 1 kg, microharness was shown to be near 350 $HV_{0.05}$ instead of near 250 HV_1 typically, due to the involvement of porosity for the latter. Moreover, for both loadings, microhardness was shown to be higher in coatings than in loose particles (about 192 $HV_{0.05}$ for the latter).

Coating-Substrate Interface properties.

Interface Soundness. Quantitative image analysis of optical images over a coating-substrate interface length of 20mm was carried out. Laser substrate surface pre-treatment could be beneficial, whatever laser conditions (despite no test had been carried out at 75 Hz), except for the most severe conditions (i.e. high density coupled with a high pulse rate) for the Ni-based system (Fig. 8).

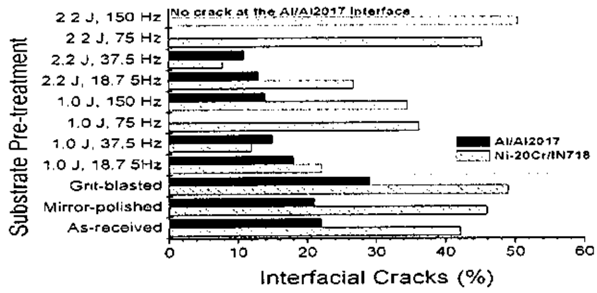


Fig. 8. Crack percentage at the coating-substrate interface for the 2 materials systems.

Laser effect was significant, although grit blasting was detrimental rather surprisingly, one may, however, assume to be partly due to alumina grit inclusions at the interface. In the best conditions, there were very few cracks only for the Ni-based system or no crack at all for the Al-based system (Fig. 9).

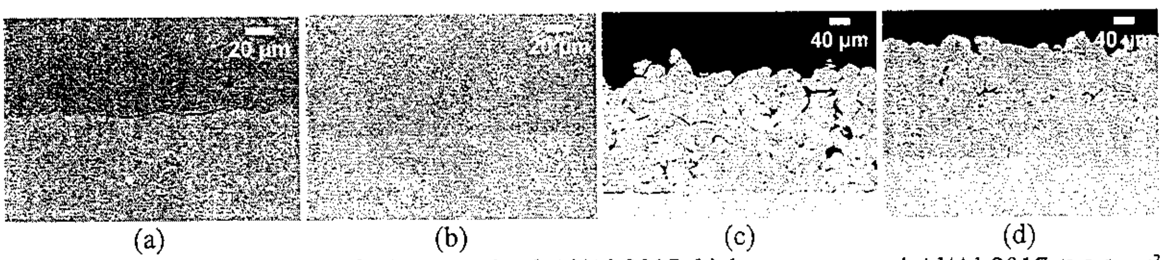


Fig. 9. SEM cross-sections of a) as-received Al/Al 2017, b) laser-processed Al/Al 2017 (2.2 J.cm^{-2}, 150 Hz), c) as-received Ni-20Cr/IN718, d) laser-processed Ni-20Cr/IN718 (2.2 J.cm^{-2},37.5 Hz).

For the Ni-based system, to the difference of what was encountered for the Al-based system, there was an optimum for laser conditions. Above a certain energy density and/or pulse rate, one may assume too a high melting of the substrate surface occurred. This should provoke pores and cracks from change in the interaction between the impinging particles and the substrate.

In contrast, when using the best laser conditions, tomography ascertained that coating-substrate interface was very sound. No crack could be detected actually at the interface as porosity could be in the coating, despite a high resolution (Fig. 10). Moreover, the interface remained fairly flat.

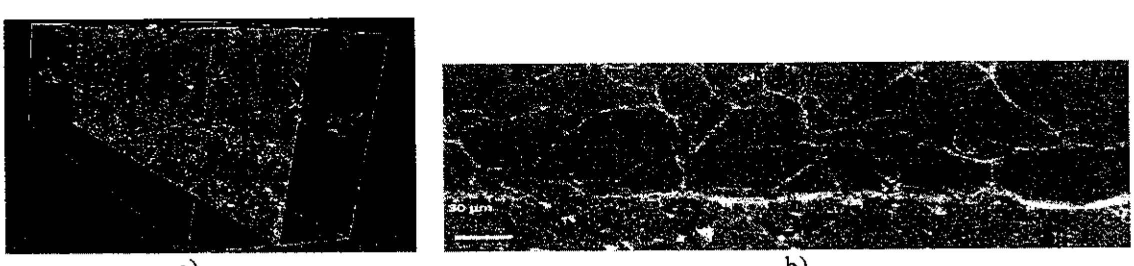
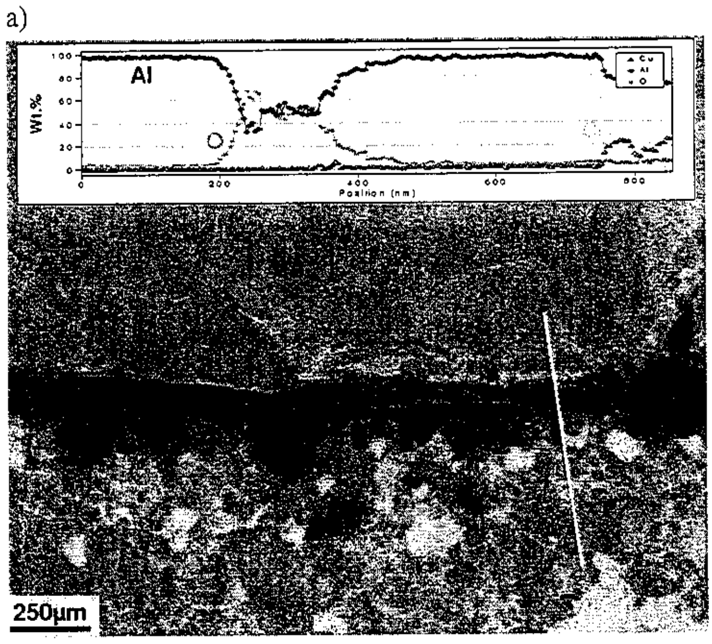


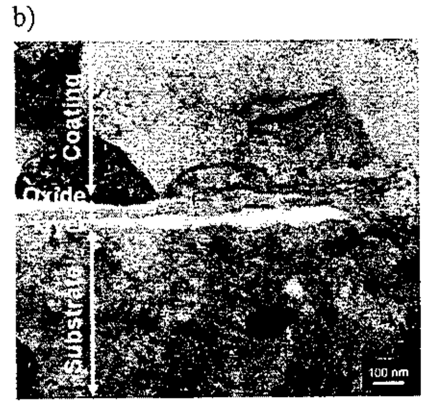
Fig. 10. Tomographic images of Al/laser – processed Al 2017 (2.2 J.cm^{-2}, 150 Hz), a) 3D image of porosity in a parallelepiped of 358x273x120 µm^3 in size), b) 2D scan showing the interface.

Fine-scale features. A typical oxide layer of about 100nm in thickness could be observed at the coating-substrate interface in cold-sprayed "as-received" Al 2017. This oxide layer did exist prior to coating as usual onto Al-based materials. In contrast, the interface for laser-processed Al 2017, was free of oxide due to efficient and stable (for the time before coating at least) cleaning of the substrate (Fig. 11). Two types of oxides (in gray in Fig. 11a) did exist. One was amorphous and made of Al oxide, the stoichiometry of which was not that of alumina. The other one, as part of the substrate surface, was crystalline alumina. Most of cracks propagated at the 2-oxide boundary.

a)



b)



c)

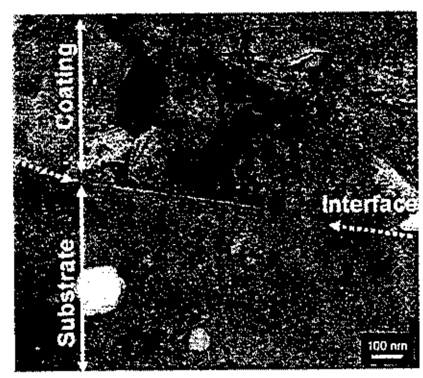


Fig. 11. TEM images of, a), b) Al/as-received Al 2017, and c) Al/laser- processed Al 2017 (2.2 J.cm^{-2}, 150 Hz).

a) HAADF image + EDX profiles along the white line, b) and c) BF images

TEM analysis of interfaces for the Ni-based system is in progress. Despite there was no result yet, one may assume the improvement from laser pre-treatment was more due to thermal effects than to physico-chemical effects as in the Al-based system.

Adhesion. LASATesting of Al-coated unprocessed and laser-processed Al 2017 showed a much better adhesion for the latter, due to the removal of the oxide layer by the laser beam prior to cold spray. The tensile stress at the interface, σ_{22}, was calculated from modeling/numerical simulation of 1D and 2D shock wave effects within the coating-substrate system and averaged over the whole laser spot [14]. Adhesion strength could therefore be determined from "post-mortem" observation of interfacial cross-sections and study of VISAR velocity profiles (profiles could be seen in [9]) during the test, since both of which showed when coating debonded (Fig. 12). It was shown to be above 629 MPa but below 681 MPa for laser-processed Al 2017 compared to below (one may assume much below) 562 MPa for as-received Al 2017. Further tests at other laser energy density levels are in progress, including for the Ni-based system, to narrow the range for determination of the adhesion strength. Incidentally, for the Al-based system, LASATesting at 629 MPa, i.e. at 2.6 GW.cm^{-2}, showed that the Al-Al interface (particle-to-particle bond strength) was weaker than the Al-Al2017 interface due to cracks within the coating rather than at the coating-substrate.

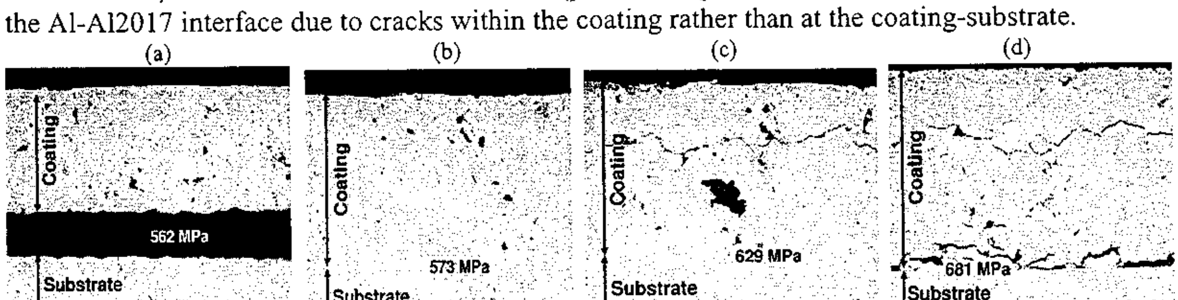


Fig. 12. Cross-section optical images of LASATested cold-sprayed Al onto, a) as received, b), c) and d), laser-processed (2.2 J.cm^{-2}, 150 Hz) substrate, when tested at various interface stress levels. Inserted: σ_{22} at the interface.

Conclusion.

This study demonstrated that, in cold spray, direct coupling of a laser head to the cold spray gun could be very beneficial for coating adhesion. A significant improvement was exhibited for Al-based and Ni-based coating-substrate systems. This resulted from oxide cleaning and/or heating of the substrate prior to coating build-up, depending on the involved materials and laser pre-treatment conditions.

LAser Shock Adhesion Test, namely LASAT, was shown to be powerful to determine adhesion strength accurately; which could be interpreted through TEM and quantitative image analyses of the corresponding coating-substrate interfaces.

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