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Selective single pulse femtosecond laser removal of alumina (Al_2O_3) from a bilayered Al_2O_3/TiAlN/steel coating

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ABSTRACT

We studied surface modification of a double layer protective coating on steel induced by single fs laser pulse irradiation in ambient air. The outer alumina (Al_2O_3) layer, which protects against aggressive environments, was 1.7 μm thick and the titanium aluminum nitride (TiAlN) layer in contact with the steel surface had a thickness of 1.9 μm. The pulses ($\lambda = 775$ nm, $\tau = 200$ fs) were generated by a Ti:sapphire laser source. The pulse energy was varied from 0.32 μJ to 50 μJ, corresponding to an incident laser fluence of 0.11 J cm^{-2} to 16.47 J cm^{-2}. The surface damage threshold was found to be 0.20 J cm^{-2} and the alumina layer removal was initiated at 0.56 J cm^{-2}. This selective ablation of alumina was possible in a wide range of fluences, up to the maximum applied, without ablating the TiAlN layer beneath.

1. Introduction

Alumina ceramic (Al_2O_3) is used in a wide range of applications, from engineering to medicine, mainly in its bulk form. Due to its favorable physical properties like high hardness, thermal stability, dielectric strength, low wear, chemical and radiation resistance, alumina is also applied as a protective coating [1]. Titanium nitride (TiN) is a well known protective surface coating for steel in many applications. Titanium–aluminum based nitride (TiAlN) obtained by incorporation of aluminum in TiN possesses excellent hardness and thermal stability [2], and a significantly enhanced oxidation resistance. A bi-layered coating composed of dielectric alumina and metallic nitride ceramic (Al_2O_3/TiAlN) deposited on steel exhibits excellent physical and chemical characteristics, which combine good properties of both coating materials [3]. Conventional mechanical micro-structuring of hard and brittle materials is difficult, and applying focused laser pulses is often the sole solution. In contrast to longer laser pulses of several nanoseconds, it has been demonstrated that short ps laser pulses or ultra-short fs pulses can be easily adjusted to induce small or no collateral damage and/or thermal effects in solid materials [4,5]. Ultra-short laser pulse modification of hard coatings is valuable for applications in nanotechnology, microelectronics, tribology, among many others [6]. Selective ablation in the form of regularly distributed micro holes could be useful, for example, as “reservoirs” for solid lubricant in tool manufacturing [7], for precision micro-structuring in electronics, and so on. Results on bulk alumina processing by femtosecond laser pulses have been reported [8,9], but few data are available on femtosecond laser interaction with alumina in the form of coating [10].

To the best of our knowledge, the laser damage threshold (D_{th}) with fs laser pulses has been determined for bulk alumina samples [8,9] and for thick alumina coating [10]. This information served as a guide for the present investigation, but the main objective was the study of the outcome of the interaction between a typical fs laser pulse with a bi-layered coating when the top layer is practically transparent to the laser wavelength in the linear absorption regime (its linear absorption coefficient is 30–70 cm^{-1}).

2. Materials and methods

The laser irradiated target was an ASP 30 high speed steel sample, top coated by reactive sputtering with an Al_2O_3/TiAlN bilayer in a commercial installation (CC800, CemeCon), equipped with four 8 kW unbalanced magnetron sources [11]. The Al_2O_3 coating was applied on steel previously coated with TiAlN. Total thickness was 3.6 μm, with the top alumina layer of 1.7 μm and the underlying TiAlN layer of 1.9 μm (Fig. 1). According to an XRD analysis, the alumina layer mainly consisted of the polycrystalline χ-Al_2O_3 phase.

We used for irradiation a Ti:sapphire laser source equipped with a chirped pulse amplification system, model Clark-MRX 2101 [12]. The wavelength of the output beam was 775 nm, with a pulse duration of $\tau_p = 200$ fs. The spatial profile of the beam was nearly Gaussian. The linearly polarized beam was concentrated with a lens (75 mm focal

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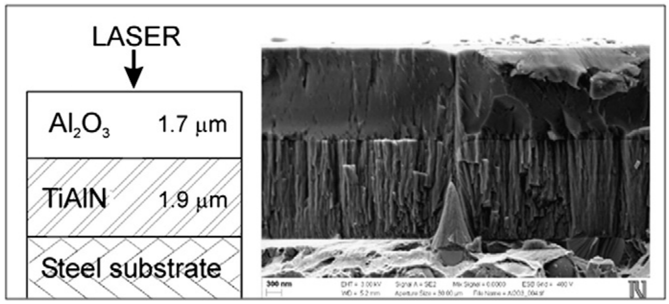

Fig. 1. Left side: schematic cross-section of the laser target with layers thickness and laser beam direction indicated. Right side: SEM micrograph of a cross-sectional break (as an illustration).

length) and directed perpendicularly to the target surface. Sample irradiation was carried out in air with single fs laser pulses whose energy E_p ranged from 0.32 μJ to 50 μJ.

For a Gaussian spatial beam profile with a $(1/e^2)$ beam half-waist w_0, and for pulse energy E_p, the maximum laser fluence at the target is $F_0=(2E_p)/(\pi(w_0)^2)$ and peak power density $I_0=F_0/\tau_p$. In our experiments the beam half-waist was $w_0=13.9$ μm, so the corresponding fluences ranged from 0.11 J cm^{-2} to 16.47 J cm^{-2}, and peak power density from 0.55 TW cm^{-2} to 82.35 TW cm^{-2}.

For better statistics, the irradiation was performed in a matrix of 9 (3×3) spots with the same pulse energy E_p (Fig. 2).

Surface morphology after irradiation was examined by optical microscopy, scanning electron microscopy (SEM), and non-contact 3D profilometry. Some typical examples are shown in Figs. 3 and 4. The SEM was coupled to an energy-dispersive analyzer (EDX) for determining surface local composition. Non-contact 3D surface profilometry was applied to map-out the geometry of the ablated/damaged area.

3. Results and discussion

The D_{th} value was determined as the lowest fluence that causes irreversible surface modification, and was found to be 0.20 J cm^{-2}. The damage appeared in the form of a doughnut shaped bulge on the surface of the top alumina layer. Apparently, it is a consequence of strains generated at the interface between the layers, or a plastic deformation of the top layer itself. This distorts the top layer and produces the bulge on its surface. An illustration is given in Fig. 3 with two images of the same spot in two different electron scattering modes. Surface swelling and/or plastic deformation at sub-melting conditions as a precursor to ablation has been observed before [13,14].

The obtained D_{th} value is lower than the one reported for the bulk Al_2O_3, which varies from 1.1 to 5.62 J cm^{-2} depending on the crystallographic status and the morphology of the sample [8–10].

At higher fluences, the alumina layer was completely removed from an area that depended on fluence, exposing the remaining TiAlN layer underneath. The threshold fluence for this selective removal was found to be 0.56 J cm^{-2}.

A laser pulse of 0.56 J cm^{-2} fluence and above (Fig. 4) completely removes the top alumina layer, but does not modify the underlying TiAlN layer in any way, even at the highest fluence used, 16.47 J cm^{-2}. This was confirmed both by morphological (SEM) and chemical (EDX) analyses. The SEM micrographs show the same surface features of the spots, regardless of the laser fluence that produced them (Fig. 4). The features belong to the exposed TiAlN surface, with characteristic lumps that replicate the look of the steel surface beneath this layer, with its carbide lumps that are left after completing the polishing of steel.

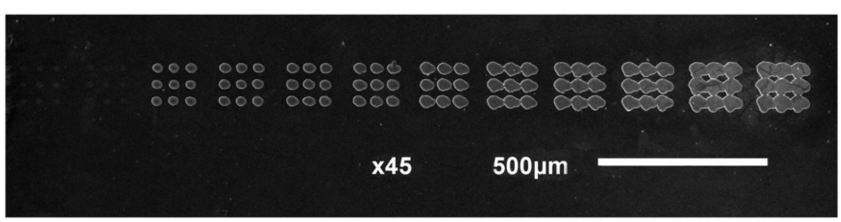

Fig. 2. SEM micrograph of the sample surface after irradiation. 12 matrices of 3×3 spots are visible, corresponding to laser pulse irradiation energies of 0.9, 1.2, 1.7, 2.7, 3.6, 5, 10, 15, 20, 25, 37.5, and 50 μJ.

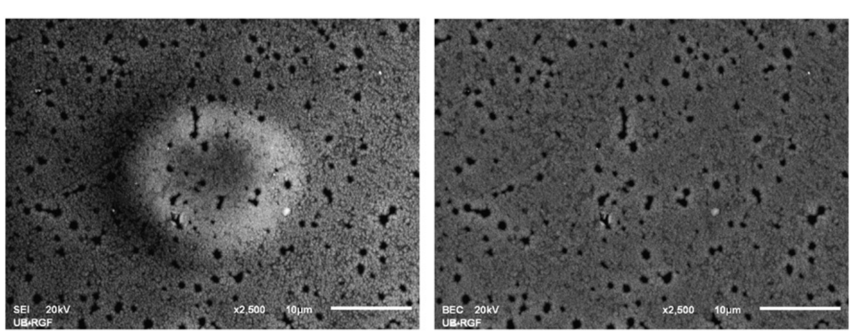

Fig. 3. SEM/micrographs of the same "bulge" produced by a pulse of 0.30 J cm^{-2}, recorded with normal electron scattering (left), and backscattering electrons (right). No "bulge" is visible with backscattering electrons, i.e. there is no change in the composition of the surface, which confirms that the top alumina layer is unbroken.