SAND94-0042C

Conf-9405116--1

Ion Beam Surface Treatment: A New Capability For Rapid Melt And Resolidification Of Surfaces

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ABSTRACT

The emerging capability to produce high average power (5-250 kW) pulsed ion beams at 0.2-2 MeV energies is enabling us to develop a new, commercial-scale thermal surface treatment technology called Ion Beam Surface Treatment (IBEST). This new technique uses high energy, pulsed (≤100 ns) ion beams to directly deposit energy in the top 2-20 micrometers of the surface of any material. The depth of treatment is controllable by varying the ion energy and species. Deposition of the energy with short pulses in a thin surface layer allows melting of the layer with relatively small energies and allows rapid cooling of the melted layer by thermal diffusion into the underlying substrate. Typical cooling rates of this process (10^9-10^{10} K/sec) cause rapid resolidification, resulting in the production of non-equilibrium microstructures (nano-crystalline and metastable phases) that have significantly improved corrosion, wear, and hardness properties.

We have conducted IBEST feasibility experiments with results confirming surface hardening, nanocrystaline grain formation, metal surface polishing, controlled melt of ceramic surfaces, and surface cleaning.

INTRODUCTION

Recent advances in high average power, pulsed ion beam systems are enabling a new technology to achieve rapid melt and resolidification of surfaces. Researchers at Sandia National Laboratories and Cornell University have developed the capability to produce 5-250 kW average power pulsed ion beams at 0.2-2 MeV energies using a repetitively pulsed (up to 120 Hz) concept designed for long component lifetimes. This new capability is enabling us to develop a commercial-scale thermal surface treatment technology called Ion Beam Surface Treatment (IBEST). This new technique uses high energy, pulsed (typically ≤100 ns) ion beams to directly deposit energy in the top 2-20 micrometers of the surface of any material. The depth of treatment is controllable by varying the ion energy and species. Deposition of the energy in a thin surface layer (Figure 1) allows melting or vaporization of the layer with relatively small energies (1-10 J/cm^2 for metal surfaces) and allows rapid cooling of the melted layer by thermal diffusion into the underlying substrate. Solidification of metals at the cooling rates typical of this process (10^9-10^{10} K/sec) results in the production of non-equilibrium microstructures (nano-crystalline and metastable phases) in the surface layer. Experiments with both laser and ion beams[1-8] have shown that surfaces produced by this rapid thermal quenching have significantly improved corrosion, wear, and hardness properties.

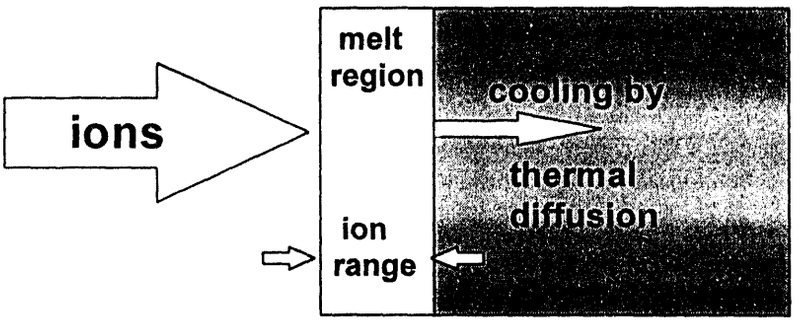

Figure 1. Ion BEam Surface Treatment (IBEST) uses a pulsed, high energy (0.2-2 MeV) ion beam to deposit energy over the classical ion range, typically 2-20 microns, in a surface, raising its temperature to melt. Thermal diffusion rapidly (10^9-10^{10} K/sec) cools the surface, leading to the formation of amorphous layers and the production of non-equilibrium microstructures by rapid quenching.

Ion Beam Surface Treatment (IBEST) is a thermal process that does not significantly change the atomic composition of the sample. The ion pulse rapidly heats a thin surface layer to melt using typically only 3×10^{13} ions per pulse. Over the ion range the implanted ion concentration is less than 10^{-3}atomic percent. The short pulse length allows the heated depth to be confined to approximately the ion range by limiting the effect of thermal diffusion. Thermal diffusion lengths in 60 ns are 1 and 4 microns in stainless steel and aluminum respectively, less than the proton range in the materials at typical IBEST ion energies of 0.4-1 MeV. The use of a new Magnetically-confined Anode Plasma (MAP) ion beam system[11-14] described later allows any gas ion to be used to deposit energy in materials. Protons, having the largest range in materials, can provide relatively deep treatment ranging from 5-15 microns in aluminum for energies of 0.5 to 1 MeV respectively.

This work was supported by the United States Department of Energy under Contract DE-AC04-94AL85000.

DISCLAIMER

The effects of Ion Beam Surface Treatment are similar to surface treatment using pulsed lasers but IBEST technology provides unique capabilities that allow it to avoid many problems intrinsic to laser technology including energy coupling to metals, edge effects, and high cost due to its in-depth energy deposition, large energy per pulse, and the low capital cost of hardware.

The energy coupling of ion beams to a material is independent of the surface preparation and only weakly dependent on the actual material. The availability of a clean, single species ion source opens up new areas for metal studies, especially in the solidification behavior of simple alloys. Thermal quench rates and the corresponding solidification velocities can be controlled by varying the beam energy and the ion species.

The typical area treated by a single IBEST pulse ranges from 100-1000 cm^2 depending on the application. This capability and our new repetitive pulse technology are key elements enabling high volume commercial applications.

Results Of Initial Experiments

IBEST experiments have been performed on several facilities including Sandia's Repetitive High Energy Pulsed Power (RHEPP) facility, Cornell University's LION accelerator, and LANL's Anaconda accelerator. These experiments are supported by an integrated team of researchers in pulsed power, beam physics, and materials science from Sandia National Laboratories, Cornell University, Los Alamos National Laboratories, and the UNM/LANL/SNL Advanced Materials Laboratory. Single pulse and bust-mode tests at 1/3 Hz have been used to produce initial treated samples while hardware for full scale repetitive operation is being optimized. Results from initial analysis confirm surface hardening, amorphous layer and nanocrystaline grain size formation, metal surface polishing, controlled melt of ceramic surfaces, surface cleaning of hydrocarbon layers from 304 stainless steel, oxide layer removal, and corrosion resistance.

These initial experients clearly demonstrate the ability of Ion Beam Surface Treatment to significantly modify the properties of materials. The ion source for all of these initial experiments was a "flashover" source which produces a mixed species ion beam. Other experiments[15] at Cornell University using this ion source have indicate that these beams are made up of approximately half H^+ ions and half heavier ions, predominantly C^+ and C^{++}. Future experiments will be done using the new, single species, Magnetically-confined Anode Plasma ion source described later in this article. The following sections describe some of our initial experiments in more detail.

Treatment O-1 tool steel

These samples were treated using Cornell University's LION accelerator (1 MeV, 4Ω, 40ns FWHM). The ion energy during the FWHM of the power pulse varied from approximately 0.5-1 MeV. The ion energy delivered to the surface was approximately 10 (+/- 30%) J/cm^2 as measured by biased and apertured ion collectors and the load voltage monitor. The samples were located approximately 25 cm from the beam system. Treatment was done at a vacuum level of approximately $2x10^{-4}$ torr.

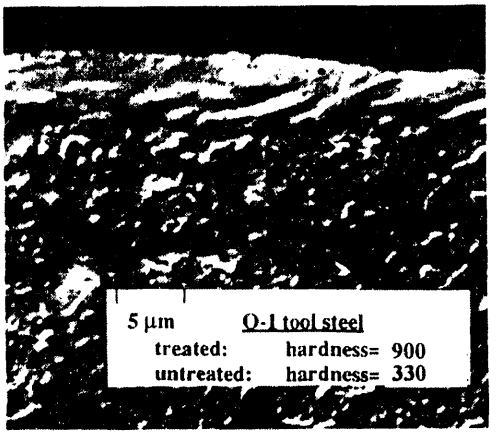

Figure 2. This cross sectional view of an O-1 tool steel sample shows the effects of rapid surface melting and cooling by a 50 ns, 10 J/cm^2, 0.5-1 MeV mixed proton and carbon beam.

Cross-sections of IBEST-treated O-1 tool steel samples were examined[18] using an optical microscope as well as cross-sectional and plan view Transmission Electron Microscopy (TEM). An optical micrograph of the cross-section of an IBEST-treated sample is shown in Figure 2. The near-surface several microns of the sample were featureless. In contrast,

the underlying untreated tool steel material had an equilibrium structure composed of α-Fe (bcc-iron) and large iron carbides. The results of the optical metallography evaluation suggested that the iron carbides in the treated region had been largely redissolved into the bcc-iron matrix. The TEM examination of the IBEST-treated O-1 tool steel sample revealed that the near surface region of the sample was composed of microcrystalline grains approximately 20 nm in diameter. Hardness testing on the O-1 tool steel samples was performed using a Shimadzu microindentation hardness tester. Knoop indentations were made using a 25 gr load on the knoop tip yielding a Knoop hardness (H_k) of H_k = 900 for the treated surface and H_k = 330 for the untreated surface of O-1 tool steel.

Both the optical metallography and TEM results indicate that carbon was dissolved into the Fe matrix during the pulsed beam treatment. The kinetics of the iron carbide dissolution process during heating, melting, and resolidification were apparently more rapid than the kinetics required for carbide reprecipitation during cooling. The presence of FeO in the treated layer suggests that oxide was incorporated into the layer from the oxidized, untreated surface when melting occurred during treatment. Oxygen may also have been incorporated during melting from the background gasses in the treatment chamber.

Polishing of Ti-6Al-4V

In other experiments we treated Ti-6Al-4V on the Anaconda accelerator (400kV peak voltage, 40 kA total current, 500 ns pulse duration) at a treatment level of 7 J/cm^2 (+/- 30%) at 250-400 keV using a 400 ns ion pulse. The surfaces were treated using 4 pulses separated by at least 5 minutes between pulses. The untreated and treated surfaces are shown in Figure 3. The surface roughness of the untreated, machined surface was approxmately 5 microns. IBEST treatment resulted in a reduction of roughness to 0.1 micron. The energy deposited in the top 3-4 microns of the near surface region in these experiments was more than sufficient to raise the temperature to the melting point and was likely large enough to cause some ablation of the surface. The time the surface was above the melting point can be roughly estimated from the energy deposition profile and the calculated thermal diffusion properties of the material to be 250-500 ns.

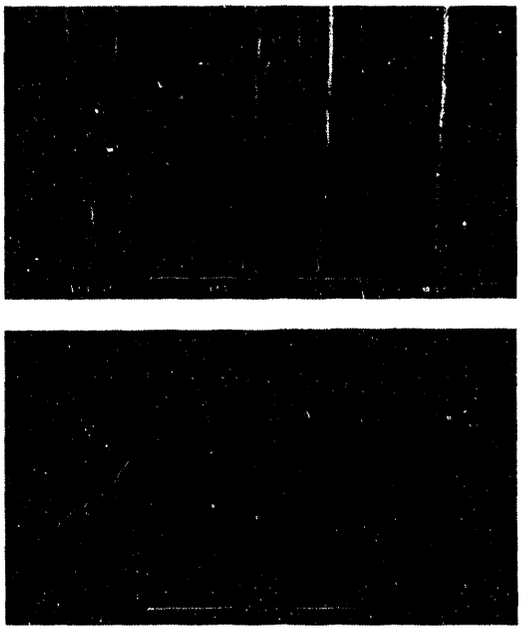

Figure 3. IBEST treatment of a Ti-6Al-4V machined surface (top) treated with 4 pulses of a 400 ns duration, 7 J/cm^2 mixed proton and carbon beam demonstrates significant surface smoothing to a 0.1 micron scale roughness.

Corrosion resistance

Initial corrosion resistance studies have been performed on the RHEPP facility at Sandia National Laboratories. In this work, samples were treated using a 700 keV, 60 ns, mixed proton-carbon beam at 2-3 J/cm^2. Corrosion resistance of the treated aluminum alloy surfaces has been assessed by electrochemical testing and by salt spray exposure testing. Treated alloys tested thus far include 2024-T3 (Al-4.4Cu-1.5Mg-0.6Mn), 6061-T6 (Al-1.0Mg-0.6Si), and 7075-T6 (Al-5.6Zn-2.5Mg-1.6Cu). Electrochemical tests used include anodic polarization and electrochemical impedance spectroscopy (EIS) conducted in an areated aqueous 0.5M NaCl solution. Exposure testing has been conducted at controlled temperatures in a

saturated salt fog environment per ASTM B117. Anodic polarization of 2024-T3 has shown that passive current densities are reduced and pitting potentials are shifted to more positive potentials indicating improved resistance to localized corrosion. Figure 4 illustrates these results.

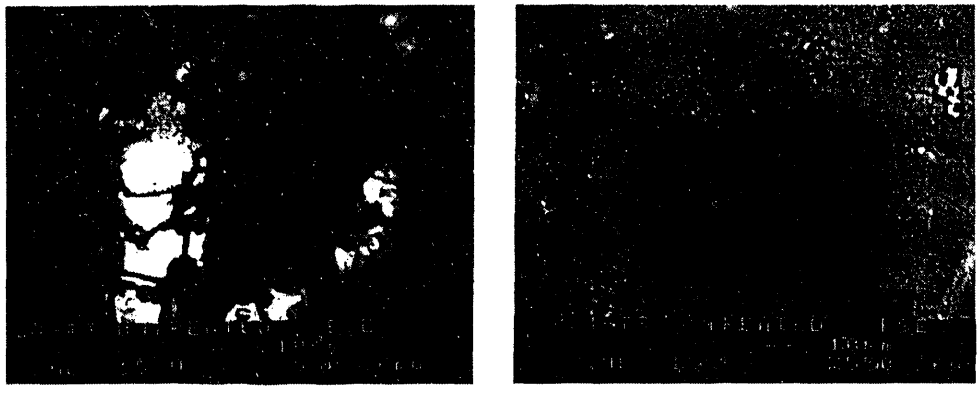

Figure 4. A 168 hour salt fog test (ASTM B117) of untreated and treated samples of Aluminum 2024-T3 shows resistance to pitting due to IBEST treatment.

Controlled melt and resolidification of alumina surface

On the LION accelerator at Cornell University we treated a polished Al_2O_3 sample with a single pulse at a level of 10-20 J/cm^2 to demonstrate controlled melt and resolidification. The result is shown in Figure 5. This technique shows promise for surface porosity reduction but also shows some microcracking on a 0.1 micron scale.

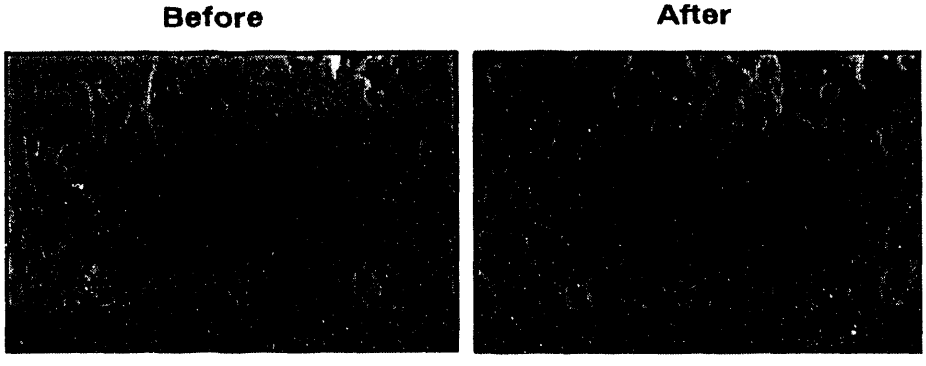

Figure 5. This alumina sample was treated using a single pulse, 0.6-1 MeVmixed proton and carbon beam at 10 J/cm^2. The result shows controlled melt and resolidification of the ceramic surface without serious problems although some 0.1 micron scale cracking was observed. The width shown is 100 microns.

Enabling Technologies For IBEST

Until recently pulsed ion beams have not been considered a viable technology for routine materials processing applications because of their inability to deliver the multi-kilowatt average powers with long component lifetimes needed for commercial processing applications. During the past few years there has been significant progress in two complementary technologies that now enable the design of 5-500 kW average power, >10^8 shot lifetime ion beam surface treatment systems for materials processing.

The first of these advances is the development of a compact, electrically efficient, repetitively pulsed, magnetically switched pulsed power system capable of 10^9 pulse component lifetimes. This prototype system, the Repetitive High Energy Pulsed Power (RHEPP) facility[10] (0.8 MV matched voltage, 35Ω, 60 ns FWHM pulse duration, 120 Hz repetition frequency), began operation this year at Sandia National Laboratories. This new facility, the first of its kind in the world, is designed to operate continuously at 120 Hz, delivering 150kW average power. This system has demonstrated operation at

50% electrical efficiency from the wall plug to energy delivered to a matched load. RHEPP is also designed to allow operation at reduced pulse rates or in single pulse mode if desired. Its capability to efficiently produce high average power, high voltage electrical pulses using a compact design is a breakthrough for the commercial application of pulsed power.

The second advance is an ion beam system that is capable of operating repetitively and efficiently to transform the pulsed power of RHEPP into an ion beam. An ion beam system capable of operating at repetitive pulse rates of 100 Hz in 10 pulse burst mode (active cooling was not part of the design) was demonstrated[13] at the Cornell University Laboratory of Plasma Studies. An improved version of this system is now being fielded on the RHEPP facility at Sandia for operation in burst mode. This system, the Magnetically-confined Anode Plasma (MAP) ion source, shown in Figure 6, is based on the concept of drawing ions from a single species plasma anode rather than the solid, flashover anode used in standard single pulse ion beam systems. The plasma can be formed from any gas ion.

The MAP ion beam system produces an annular beam which is brought to a broad focus symmetric about the axis shown in the figure. In the cathode (ground potential) electrode assembly (A), slow (100 μs risetime) magnetic field coils (B) produce magnetic flux which provides the magnetic insulation of the accelerating gap between the cathode and the anode (high voltage) electrode assembly (C) connected to the output of the RHEPP generator. The ion source that supplies ions to the accelerating gap is contained within the anode assembly. The MAP source operates in the following way: a fast gas valve (D) on the axis of the anode assembly produces a rapid (200 μs) gas puff which is delivered through a supersonic nozzle (E) to produce a highly localized volume of gas (F) directly in front of the surface of a fast-driving coil (G) located in an insulating support structure (H). After preionization by a 1 μs induced electric field, the fast coil is energized, inducing a loop voltage of 20 kV on the gas volume, driving a breakdown to full ionization, and moving the resulting plasma toward the flux-excluding anode field-shaping electrodes (C) in about 1.5 μs, to form a thin magnetically-confined plasma layer. The RHEPP pulse is then applied to the anode assembly, accelerating ions from this plasma layer to form the ion beam. The magnetic flux surfaces (J) at the time of beam extraction are shown. The beam propagates in vacuum to a broad focal area at the target plane at the right of the figure, where material samples are placed for treatment.

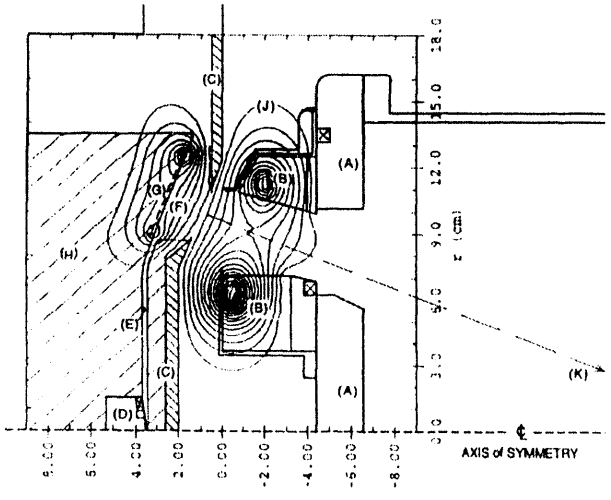

Figure 6. MAP Ion Diode for RHEPP. The cathode assembly (A) with slow magnetic field coils (B); the anode electrodes/magnetic flux shapers (C); the fast gas valve (D); the gas nozzle (E); the gas breakdown and plasma formation region (F); the fast-driving magnetic field coil (G) in the insulation support structure (H), the magnetic flux of the slow and fast coils, as they are at the time of the RHEPP accelerating pulse (J); the ion beam propagation direction to the material target (K).

Conclusions

We have demonstrated that Ion Beam Surface Treatment (IBEST) can significantly alter the microstructures of the near surface region of materials and thus the material properties. The effects of IBEST include hardening, corrosion resistance, polishing and cleaning.Initial results from these experiments indicate that IBEST can provide new capabilities for cost-effective surface treatment for a variety of applications.

Acknowledgments

Our Ti-6Al-4V smoothing experiments were conducted on the Anaconda accelerator at Los Alamos National Laboratory with the assistance of D. Rej, and W. Waganaar.

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