SURFACE AND STRUCTURAL ANALYSIS OF TRANSITION METALS

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ABSTRACT: Laser irradiation effects on transition metals have been investigated in terms of their surface modifications, changes in structural properties. Fine polished and 4N (99.99%) pure samples of copper (Cu), gold (Au) and platinum (Pt) are exposed to Q-switched Nd:YAG laser (1064nm, 10ns and 10mJ) in ambient air. Laser fluence used is 3.18 J/cm² where the focal spot size on target surface is 100 µm. A grid is formed on each sample surface imposing 200 laser shots on each spot. Radiated and un-irradiated targets are then characterized by employing Optical microscope and X-ray diffarectrometer for the analysis of surface morphology and structural properties respectively. The thermal effects are dominant in gold: also the microstructures are observed on the ablated surface of copper which shows the non-uniform behavior of heat conduction. The ablation and splashing is observed on Platinum surface. The comparison of structural changes for exposed targets reveals the change in grain size, dislocation line density and micro-strain on the target surfaces. The correlations are found between grain size, dislocation line density and strain with 2(theta) of the materials.

Key words: Laser irradiation, surface morphology, crystallography, surface hardening

1: INTRODUCTION

In many industrial applications a focused laser beam is one of the highest power density sources for materials processing because of its highly concentrated energy [1]. Material surface processing involves the ablation. When laser beam interacts with the target, it causes electronic excitations in the atoms of exposed part. These excitations force the target atoms to vibrate with higher kinetic energy and make them to collide with the neighbors atoms. In this way a cascade of collisions is generated. These cascade and sub cascades of collisions results in the temperature rise[2]. So heat affected zone (HAZ) appears around the laser exposed spot [3]. After the ablation, plasma is produced normal to the surface generating shock waves by two methods, direct ablation and confined ablation. In direct ablation plasma expands directly and shockwaves are ejected into atmosphere, whereas in confined ablation plasma expands into the material and shock waves propagate in the material [4]. Laser micromachining of different components is used in mechanical, electronic and non-intrusive medical devices. Laser induced micro sized particles are vastly used in Deoxyribonucleic acid (DNA) analysis and drug screening [5].

Laser interaction with material also results in material hardness. Hardness of metals is playing an important role in developing the new technology.

The current research work illustrates the change in surface morphology and variation in grain size of the materials due to laser irradiation.

2: EXPERIMENTATION

Fine polished and 4N pure samples of gold, copper and platinum of size $(1 \times 1 \times 0.3 \text{ cm}^3)$ are used as target material for

investigations on surface and structural analysis. The surface and structural modifications of target materials are performed by a passive Q-switched Nd: YAG laser (1064 nm, 10mJ, 1.1 MW, 9-14 ns) which has Gaussian beam intensity profile. The fluence of laser beam used is 3.81 J/cm^2 with afocal spot size is 100µm on target surface. Targets are mounted on the sample holder and are irradiated in ambient air alternatively. The laser beam is incident at an angle 0° with the normal to the sample surface. IR transmission focusing lens of focal length 8 cm is used to focus the laser beam tightly on sample. A grid is formed on the target exposing each spot for 200 laser shots.

The irradiated surface is analyzed by using Optical microscope (OLYMPUS STM6-LM, Corporation) of resolution 0.1µm at 1000x magnification. The PANalytical X^v pert PRO super X-ray diffractometer with CuK α radiation ($\lambda = 0.15418$ nm) is used for structural investigations. The XRD tube operates at 40 kV with a filament current 30 mA. The 20 (Bragg's angle) angular regions between 10° and 90° are recorded at a scan rate of 0.05 mVs⁻¹.

3: RESULTS AND DISCUSSION

Surface morphological changes due to exposure of metals are analyzed by Optical micrographs at 10X magnification (figure 1). Some significant changes occur in heat affected zone (HAZ) [6, 7]. The non-uniform shape of HAZ shows the non-symmetric heat conduction. Also the presence of impurities is responsible for such non-uniform damage. Micro sized particles and splashed material are observed outside the heat affected zone (HAZ) fig 1(c). These processes are thermal in nature.



Figure 1: Optical micrograph (10x) of irradiated (a) gold (b) copper (c) platinum, for 200 laser shots



Figure 2:A plot of Bragg's angle 2(theta) vs (a) diffracted x-ray intensity (b) dislocation line density (c) grain size (d) strain, for unirradiated and irradiated gold



Figure 3:A plot of Bragg's angle 2(theta) vs (a) diffracted x-ray intensity (b) dislocation line density (c) grain size (d) strain, for unirradiated and irradiated copper

When laser energy is converted into thermal energy, melting takes place which leads towards material ablation. Heat compensation between irradiated hot region and cool surroundings shows a material re-solidification with sharp boundaries. The micrograph for gold presented in figure 1(a) indicates a non-uniform thermal energy transfer at the surface. Micro cracks production on the gold surface is due to the shock waves generation on the target surface. In copper (figure 1b), material is ablated and the heat is conducted nonsymmetrically around the crater due to which the shape of the crater is non-uniform. The uniformity of Heat-diffusion is associated with the propagation of shock waves into the material. These shock waves damage nearby structures or delaminates multilayer materials [8]. The production of shock waves directly depends upon the energy deposition on the material surface. Surface morphology of platinum indicates the crater formation due to intense beam and particles splashed out in outward direction from ablated part of the material (figure 1c). The heat waves that propagate through the HAZ produce mechanical stresses and generation of micro cracks in the surrounding of the material surface [9].

The crystallographic or structural changes are examined using X-ray diffractometery. In figure 2(a) gold shows a homogenous structure and crystallanity. Intensity of diffracted X-rays increases for all the planes. A maximum intensity increase occurs for plane (111). Structural imperfections produced due to thermal stresses developed by repetitive laser shots affect directly the grain size of material. Grain size decreases for the plane (111), (200), (220) and (222) as illustrated in figure 2(b).The consequent changes in dislocation line density and strain are shown in figure 2(c-d). Un-irradiated copper shows a homogenous and crystalline behavior. After exposure of energetic laser beam there is an increase in diffracted x-ray intensity as shown in figure 3(a). The atoms of exposed part displace from their initial positions [10]. Reorientations of atoms from their initial lattice sites cause the decrease in grain size of the exposed target (figure 3b) and correspondingly an increase in dislocation line density and strain values as indicated by figure 3(c-d).

Grains sizes and diffracted x-ray intensity both vary due to heating, recrystallization and defects generated due to laser irradiation which leads to imperfection in structure of platinum. A large grain breaking into smaller ones reduces the grain size, thus an increase in the dislocation density [11,12]. On the other hand in Figure 4(a) very large change in intensity appears for plane (111). When diffusion occurs through the grain boundaries, grains also merge to make larger grains, so decreasing the length of the grain boundaries. During the cooling of material there is a big change in grain size for (111) plane of platinum as shown in figure 4(b).



Figure 4:A plot of Bragg's angle 2(theta) vs (a) diffracted x-ray intensity (b) dislocation line density (c)grain size (d) strain, for unirradiated and irradiated platinum

Grain size variations leads to changes in dislocation line density and strain in the materials, and displacement of planes from its original position to modify the hardness of the materials. Variation in hardness greatly depends upon the grain size, dislocation line density and strain produced.

4: CONCLUSIONS

The gold, copper and platinum samples are irradiated with Nd:YAG laser for 200 laser shots in ambient air. Micro cracks are produced on the platinum and gold surfaces. Crater is formed on the surface of copper. The prominent change in grain size occurs in most dense plane (111). The decrease in grain size leads to increase in strain on the plane, it mean that stresses has inverse relation with grain size.

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