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Prospects for using high power x-rays as a volumetric heat source

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ABSTRACT

Third-generation, high-intensity, x-ray synchrotron radiation sources are capable of producing high heat-flux x-ray beams. In many applications finding ways to handle these powers is viewed as a burden. However, there are some technological applications where the deep penetration length of the x-rays may find beneficial uses as a volumetric heat source. In this paper we discuss the prospects for using high power x-rays for volumetric heating and report some recent experimental results. The particular applications we focus on are welding and surface heat treatment. The radiation source is an undulator at the Advanced Photon Source (APS). Results of preliminary tests on aluminum, aluminum metal matrix composites, and steel will be presented.

Keywords: x-rays, insertion devices, welding, heat treatment, volumetric heating

1. INTRODUCTION

Recent years have seen the development of synchrotron radiation (SR) sources for a number of novel applications. For example, the potential of x-rays for sub-micron parallel processing has given rise to the field of x-ray lithography and the deep penetration properties of high energy x-rays has made possible the development of micro-machining. The advent of third-generation, high-power, insertion-device (ID) based SR sources offers the possibility of utilizing x-rays for another novel type of materials processing - localized, volumetric heating.

X-ray beams from IDs are capable of producing heat fluxes of greater than 10^4 watts/cm² and several kilowatts of total power. Such high power beams are capable of melting or even vaporizing a wide variety of materials. Managing such high powers has presented challenges to designers of modern-day SR accelerators and beamlines. However, since the x-rays in the range 3-200 keV have penetration lengths on the order of 0.001 to 50 mm for a number of materials (see Fig. 1(a)), they have the potential for volumetric heating. The applications that we will focus on in this paper are welding and surface transformation hardening.

Presently the most common welding techniques are tungsten inert gas (TIG), metal inert gas (MIG), resistance, electron beam (EB), and laser beam (LB).¹ Of these, only the last two are capable of producing localized, full penetration, autogenous (without filler wire) welds and heat treatments. Since the penetration depth of these two sources is relatively shallow (1-3 μ m), the only way to fully penetrate thicker samples was to literally bore a "keyhole" through the material, using power densities in excess of 10^6 watts/cm². For many classes of materials this is not a severe drawback and does little to hamper the mechanical strength of the joint. However, for certain types of materials, such as ceramics and metal-matrix composites (MMCs), such processing can be catastrophic, leading to partial or complete failure at the interface.² MMCs are composed of a metal (usually Ti or Al) within which a reinforcing ceramic (usually Al₂O₃ or SiC) particulate, whisker, or fiber is imbedded. These materials are lighter and have superior mechanical and thermal properties as compared to the base metal, and are finding widespread use

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in many areas. They have great potential for use in the aerospace industry,³ provided a joining process that maintains the strength of the material can be found.

Electron beams and lasers may also be used for surface transformation hardening.⁴ For example, EBs can be used to harden components made from carbon or alloy steel. An EB directed at the component can produce a hardened surface consisting of 99.9% martensite to a depth of more than 1 mm. The EB is defocused so that it will rapidly heat, but not melt, a volume consisting of the target area to a depth determined by the range of the electrons, which is dependent on the accelerating voltage of the beam (30-150 keV). It is less straightforward to perform surface hardening with the laser. Since a defocused beam must be used to avoid melting and vaporization, the high surface reflectivity of metals at the IR wavelengths usually necessitates coating the surface with a black absorptive material, which is an additional processing operation. However, the CO₂ laser, using a focused beam, is able to melt a metallic surface since the reflectivity decreases as the temperature rises and has found many applications in hardening by surface alloying.⁵

In this paper we present results of experiments that demonstrate the potential for using high power x-rays for material processing. Measurements of Al/Al₂O₃ MMCs show that full penetration heating of a 3.3-mm-thick sample occurs with little disruption to metallurgical integrity. Experiments on 1018 cold-rolled steel indicate that significant metallurgical changes can be induced to depths of several mm. Preliminary results for other materials will also be discussed and the utility of high-energy, high power x-rays for material processing will be examined.

2. EXPERIMENTAL

The experiments were performed at the Advanced Photon Source (APS) at Argonne National Laboratory. The APS is a 7-GeV, third-generation synchrotron radiation source. The insertion device used in these experiments (undulator A) is a 72-period, 3.3-cm period length "undulator" capable of reaching a peak magnetic field of 0.849 T when the undulator gap is closed to its minimum setting of 10.5 cm, resulting in a peak x-ray heat flux of ~180 W/mm² (at the location of the experiment, 30 m from the source) for a storage ring beam current of 100 mA. At the first harmonic energy of 3.2 keV, calculations predict a spot size FWHM of 2.0 mm horizontal (h) x 1.1 mm vertical (v). As the undulator gap is increased, the energy of the first harmonic increases from 3.2 keV to 12 keV at 25 cm, while the peak heat flux decreases to 35 W/mm².

The results presented here were performed on two different beamlines. Welding trials on Al/Al₂O₃ MMCs were performed on beamline 1-ID with the undulator gap set to 11 mm, resulting in a peak heat flux of 170 W/mm² and a first harmonic energy of 3.5 keV. A fixed mask limited the beam size to 2.5 mm (h) x 1.0 mm (v). The power output from this undulator at closed gap, 100 mA of beam current, and a 2.5 mm x 1.0 mm spot is shown in Fig. 1(b). It consists of a harmonic series of quasi-monochromatic peaks; the strongest peaks lie at the odd harmonics. Although the most intense peaks are at lower energies, a considerable amount of power is at high energies, as revealed by the integrated power spectrum shown by the cross-hatched region in Fig. 1(a). This shows that only 90 watts of power is contained in the 2-20 keV region, while the remainder of the ~380 watts contained in the beam lies at higher energies. Additional welding trials on 6061-T6 Al alloy as well as heat treating studies on 1018 cold-rolled steel were performed on beamline 20-ID which also incorporated undulator A, but had a 4.5 x 4.5 mm fixed mask located at 26 m from the source to aperture the beam. This resulted in a considerably higher total power output of ~2.7 kW at 100 mA beam current and an ID gap of 11 mm.

The Al/Al₂O₃ MMC samples were obtained from MC-21, Incorporated. They consist of a 6061 Al alloy matrix reinforced with 20% calcined Al₂O₃ powder particulates. The particulate size distribution has an average size of 20.8 ± 6 μm. Samples used in this study were 25 x 100 x 3.3 mm. The 6061-T6 Al alloy (25 x 44 x 3.3 mm) and 1018 CR steel (25 x 44 x 6.6 mm) samples were obtained commercially. They

were mounted in a fixture and positioned so the x-ray beam was normal to the surface. "Bead-on-plate" spot welds were performed by irradiating a spot on the sample. The experiments were carried out in a shielded work area, in air, with a slight He purge. A schematic diagram of the setup is shown in Fig. 2.

Figure 1: (a) Absorption lengths of various materials in the energy range 2-200 keV. The cross-hatched area at the bottom shows the integrated power output of the APS undulator A, at a distance of 30 m from the source, through a 1.0 x 2.5 mm aperture, with 100 mA of beam current in the storage ring.⁶ (b) The power spectrum of undulator A under the same conditions described in (a).

Figure 2: Schematic diagram of the experimental setup. X-rays emanating from the insertion device are transported, in vacuum, down a beamline and exit into a shielded work area through a Be window where they irradiate the sample.

3. RESULTS AND DISCUSSION

The results presented in this paper were obtained on Al/Al₂O₃ MMCs, 6061-T6 Al, and 1018 cold-rolled steel. Aluminum is a relatively low-Z material and does not attenuate the power in the x-ray beam dramatically; therefore relatively little power is deposited in a 3-mm-thick sample. The steel samples are primarily composed of iron, which significantly attenuates the x-ray beam, and most of the power is deposited in the first 1 mm, for a gap of 11 mm (first harmonic energy = 3.5 keV). These facts are graphically demonstrated in Fig. 3, which shows the power attenuation for both a 2.5 x 1.0 mm and an unapertured beam. With no aperture there is considerable "wiggler" background underneath the peak structure (Fig. 1(a)), which contains a considerable low energy component. Therefore, with no aperture, a greater proportion of the power is attenuated at shorter thicknesses.

Figure 3: Power attenuation of the x-ray beam from undulator A at 11 mm gap, 100 mA current, with a 2.5 x 1.0 mm aperture (solid line, left axis) and no aperture (dashed line, right axis) as a function of penetration depth into Al (circles) and iron (triangles).

Initial experiments were performed on Al/Al₂O₃ MMCs at beamline 1-ID with the 2.5 mm x 1.0 mm aperture. Cross section SEM images of the fusion zone of a Al/Al₂O₃ MMC irradiated for ~16 s at a storage ring beam current of 91 mA are shown in Fig. 4. The entire width of the sample is shown in (a). X-rays were incident on the sample at the lower left and exited from the upper right. Although some vaporization of matrix material did occur, most of the material is still intact and the distribution of particulates stays uniform. An enlarged region of the area where the x-rays impinged on the sample is shown in (b). Note that the density of particulates is slightly higher in this region. This is probably the result of selective vaporization of the Al matrix ($T_{\text{vap}} = 2467$ C) leaving behind an excess of Al₂O₃ particulates ($T_{\text{vap}} = 2980$ C). An enlarged view of the region where the x-rays exited is shown in (c). Here the distribution of particles is uniform when compared to an unirradiated region, shown in (d).

The significance of these results lies in the fact that although melting occurred throughout the heat-affected zone, the reinforcing particulates were unperturbed. This is in stark contrast to welding trials that were attempted using both electron beam and laser sources. Regardless of sample travel speed and beam power evaluated, violent vaporization always accompanied a full penetration weld. As a result, the particulates were destroyed and the piece was cut rather than fused. Similar behavior has also been observed in laser welding of Al/SiC MMCs.⁷

Another important aspect of the results shown in Fig. 4 is the total absence of cracks in the weld zone. The matrix metal, 6061 aluminum, is notorious for solidification cracking when welded autogenously using conventional methods.⁸ Examination of our weld trials on MMCs and 6061 Al performed at low total power on beamline 1-ID showed no evidence of cracking. Weld trials were also performed on 6061-T6 Al on beamline 20-ID at a higher total power of ~2.5 kW. Microscopic examination of a cross section of the weld region did not reveal any cracks. This behavior can not be totally explained; it is conjectured that the volumetric heating results in relatively slow heating and cooling rates, which in turn places less strain on the material in the melt zone. More experiments are being planned to examine this phenomenon.

Figure 4: Cross-section SEM images of a 20% Al/Al₂O₃ metal-matrix composite sample following a bead-on-plate x-ray weld. In (a) is shown the entire cross-section. X-rays were incident on the lower left and exited from the middle-top. An enlarged view of the region where the x-rays struck the target (lower box in (a)) is shown in (b). Note the higher density of particulates in this region. In (c) and (d) are shown enlarged views of the region where the x-rays exited (upper box in (a)) and an unirradiated region, respectively. The black and white material seen surrounding the sample is from the molding compound used for mounting.

Localized steel heat-treating trials were performed on beamline 20-ID as a function of ID gap. As the gap increases, the total power decreases and the energy of the first harmonic increases. However, the critical energy decreases. The gaps settings and related parameters are shown in Table 1.

Table 1: Relevant Parameters for Steel Hardening Trials

Gap (mm)	Power (kW, 100 mA) (4.5 x 4.5 mm aperture)	First harmonic energy (keV)	Critical energy (keV)
11	2.5	3.6	26.2
14	1.4	5.4	18.9
18	0.6	8.2	12.6
21	0.3	12	9.4

Figure 5 shows the results of irradiating a 1" x 3.5" x 0.25" piece of 1018 cold-rolled steel for 2 s at 78 mA of beam current and different undulator gaps. The left side of the figure is a picture of the exposed sample while the right side shows the results of hardness scans through a cross section of the three highest power regions where localized melting occurred. The hardness scans were performed by making measurements starting at the top surface and progressing through the sample in the irradiation direction. For gaps of 14 and 18 mm, uniform hardening occurred to depths of 1-2 mm. The hardening results from rapid quenching of the liquid. This produces martensite and bainite, both of which are harder than the base metal which has a ferrite and pearlite structure. Hardening also occurs at the highest power (G = 11 mm), but appears to be less at the surface. Detailed metallurgical investigations are ongoing to determine the exact nature of the phases in the melted regions.

Figure 5:

Top: Picture of a 0.25"-thick sample of 1018 cold-rolled steel irradiated for 2 s at a beam current of 78 mA. The width of the undulator gap for each trial is shown above the spot. The scale on the bottom is in mm.

Right: Knoop hardness scans through cross sections of the irradiated regions for the three lowest gap (G) settings. The zero for the distance scale is the sample surface.

4. PROSPECTS

The results presented in this paper demonstrate the potential of using high power x-rays as a volumetric heat source for welding and surface hardening. Much further work needs to be performed in order to understand the complex metallurgical changes that occur during the process and how these changes relate to the temperature gradients produced as a result of x-ray absorption. In addition, more sophisticated apparatus needs to be developed in order to produce practical weldments. This work is ongoing. In addition to welding and surface heat treating, some other areas that may benefit from high power x-ray processing are the following.

1. Semiconductor annealing. As with lasers, x-rays could be used to anneal the damage caused by ion implantation, recrystallize doped amorphous films deposited on substrates, remove precipitates present after conventional high-temperature dopant diffusion, and diffuse surface-deposited dopant films. This could be done at lower powers and deeper depths. Furthermore, by properly tuning the energy of the x-ray beam it may be possible to selectively heat different portions of a complex device. It may also be possible to heat "buried" layers by using high energy x-rays. For instance, a thin Al overlayer will be transparent to a 15 keV x-ray but absorbed strongly by a GaAs substrate.

2. Surface alloy formation. It should be possible to produce unique surface alloys through the action of x-rays. Like lasers or electron beams they could be used to induce diffusion in ion-implanted materials. They could also be used to deposit material from the vapor while heating the surface, thus simultaneously performing chemical vapor deposition and diffusion. Tuning the photon energy above and below a core level of the substrate or the source gas should cause changes in the nature of the deposited film. It is also conceivable to irradiate a substrate through a low-Z overlayer. For instance, x-rays irradiating a low-carbon steel in contact with a carbon overlayer should induce enrichment of carbon in the surface of the steel, thereby leading to superior surface strength (case hardening).

Thermal and structural analyses need to be carried out to determine optimal beam size, beam power and heat flux, and x-ray energy (relevant to the depth of penetration) for each application. If indicated, a focused x-ray beam can be used. A compound diffractive lens (CDL)¹⁰ may be used to produce either a parallel or a converging beam. The need and the usefulness of such a focusing system for the present application is not clear at this time. However, since the CDL focuses an incident white beam at strongly energy-dependent focal spots, it may offer some promise for welding and surface treatment applications.

A mirror can also be used for focusing but because it acts as a low-pass x-ray energy filter, a good part of the incident white beam power will be absorbed by the mirror even at very small grazing angles. However, for applications where higher energy photons and deeper penetration lengths are needed, this may be desirable. Super mirrors, which can reflect a wide energy band of x-rays, may also be used, although the efficiency of such mirrors is typically low.

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