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Laser etching of austenitic stainless steels for micro-structural evaluation

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Etching is a key step in metallography to reveal microstructure of polished specimen under an optical microscope. A conventional technique for producing micro-structural contrast is chemical etching. As an alternate, laser etching is investigated since it does not involve use of corrosive reagents and it can be carried out without any physical contact with sample. Laser induced etching technique will be beneficial especially in nuclear industry where materials, being radioactive in nature, are handled inside a glove box. In this paper, experimental results of pulsed Nd-YAG laser based etching of few austenitic stainless steels such as SS 304, SS 316 LN and SS alloy D9 which are chosen as structural material for fabrication of various components of upcoming Prototype Fast Breeder Reactor (PFBR) at Kalpakkam India were reported. Laser etching was done by irradiating samples using nanosecond pulsed Nd-YAG laser beam which was transported into glass paneled glove box using optics. Experiments were carried out to understand effect of laser beam parameters such as wavelength, fluence, pulse repetition rate and number of exposures required for etching of austenitic stainless steel samples. Laser etching of PFBR fuel tube and plug welded joint was also carried to evaluate base metal grain size, depth of fusion at welded joint and heat affected zone in the base metal. Experimental results demonstrated that pulsed Nd-YAG laser etching is a fast and effortless technique which can be effectively employed for non-contact remote etching of austenitic stainless steels for micro-structural evaluation.

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1. Introduction

Metallography for micro-structural analysis is the study of underlying structure of engineering materials and it is considered as an integral aspect of material testing [1]. It aids in ascertaining product reliability by determining whether material has been processed correctly or not. Metallographic procedure involves sectioning of specimen at location of interest followed by grinding and polishing to obtain mirror finish surface. Polished surface is etched to reveal the microstructure. The most common technique for metallographic etching is selective chemical etching and numerous formulations have been used over the years [1,2]. Other etching techniques such as electrolytic etching, heat tinting, magnetic etching, gas contrasting and ion beam induced etching have also found specialized applications in various industries depending upon their suitability [2–6].

Advanced Fuel Fabrication Facility (AFFF) is presently engaged in fabrication of uranium and plutonium mixed oxide (MOX) fuel pins for upcoming Prototype Fast Breeder Reactor (PFBR) at Kalpakkam, India. A PFBR fuel pin is fabricated by loading MOX and deeply depleted uranium oxide ($DDUO_2$) pellets into stainless steel tube followed by autogenously pulsed TIG welding of tube and plug at both ends. As an integral quality control check, active metallography of tube and plug weld samples is carried for every production shift to evaluate grain size of cladding material and depth of fusion at the weld joint to ensure that integrity of pin will be maintained in the nuclear reactor till desired fuel burn up is achieved [7]. Metallography of fuel pin bearing radiotoxic material is called active metallography since the whole operation i.e., sectioning, grinding, polishing and chemical etching is done inside an enclosed containment (glove box) to prevent release of radioactivity in ambient environment [8]. But continuous use of acids such as HCl, HNO_3 and HF for etching of samples inside the glove box has resulted in corrosion at glove box joints, disintegration of inlet and outlet air filters thereby increasing risk of containment breach (Fig. 1). Furthermore, it is difficult to work in etching glove box since acidic conditions resulted in coloration of transparent glass panel and hardening of neoprene hand gloves.

All these factors have led to seek a substitute for chemical etching. Laser induced etching is pursued over other techniques due to following reasons:

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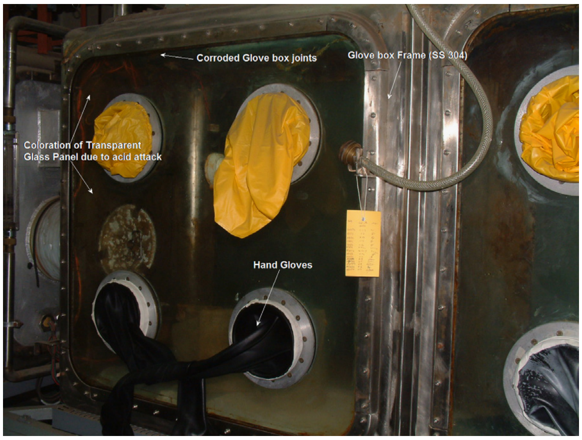

Fig. 1. Degradation of glove box structure due to continuous use of chemical reagents for etching.

- Laser etching can be done remotely since laser beam of proper wavelength can pass through glass without major loss. Laser source can be kept outside of glove box which is advantageous from maintenance point of view.
- There is no waste generation in laser etching as compared to chemical etching which is a source of alpha bearing liquid radioactive waste in active metallography. Laser etching is beneficial since storage and treatment of radioactive wastes are a major issue.
- Degradation of glove box is practically nothing since laser etching is a dry process and does not involve use of corrosive acids.

The main objective of this work is to develop laser etching system for remote etching of materials handled inside glove box. Although laser is vastly employed in today's industries for welding, cutting, drilling, micromachining and surface cleaning etc. [9,10], very little work had been carried out so far in field of micro-structural observation by laser etching. A few studies had been carried out

Table 1
Nominal composition of austenitic stainless steel used for laser etching.

Description	C	Cr	Ni	Mn	N	Ti	Mo	Fe
SS 304	0.08	18–20	8–10.5	2.0	–	–	–	Balance
SS 316 LN	0.05	17–18	12–12.5	1.6–2.0	0.06–0.08	0.05 max	2.3–2.7	Balance
SS alloy D9	0.035–0.05	13.5–14.5	14.5–15.5	1.65–2.35	0.005 max	5(%C)–7.5(%C)	2–2.5	Balance

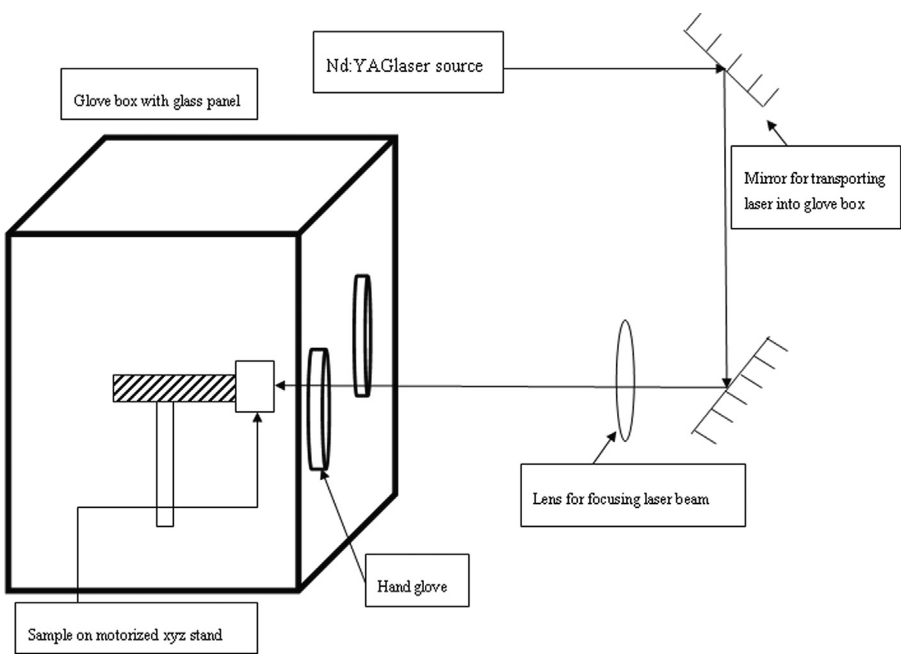

Fig. 2. Schematic for laser etching system.

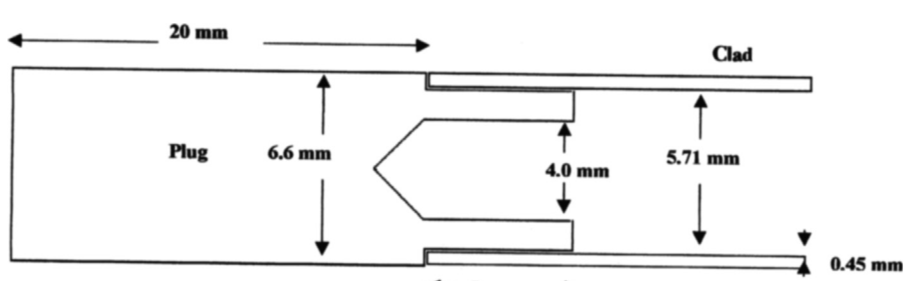

Fig. 3. Schematic of SS 316 LN plug and SS alloy D9 tube used for making PFBR fuel pin.

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