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Characterization of Nd:YAG laser ablated stainless Steel Alloys

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Abstract* - The purpose of this article is to study the influence of Nd:YAG laser on the removal of oxide from the stainless steel alloys which are used in hot sections of the aircraft engine. Oxide layer was formed on AMS 5504 & AMS 5524 sheet metals respectively by subjecting them to heat treatment. The samples were then raster scanned with Nd:YAG laser beam in the ambient air at various pulse currents and frequencies. SEM and EDS were utilized to characterize the laser irradiated surfaces. Parameters for the final cleaning trial were selected based on the results obtained from preliminary experiments. SEM images of all the laser cleaned areas showed signs of surface melting with re-cast layers formed on its surface. So, it is concluded that ablation is the mechanism responsible for removal of oxide from stainless steel alloys. A substantial decrease in oxygen % (obtained from the EDS) in the laser irradiated surfaces of AMS 5504 and AMS 5524 shows that Nd:YAG laser can effectively remove oxide from the stainless steel alloys.

***Keywords* - Stainless steel alloys, laser ablation, SEM and EDS characterization.**

1. INTRODUCTION

Stainless steel (SS) alloys are commonly used in hot section of the aircraft engine like turbine. SS alloys react with the oxygen at room temperature and elevated temperatures to form an oxide layer (Refer to Fig 1) layer over the surface.

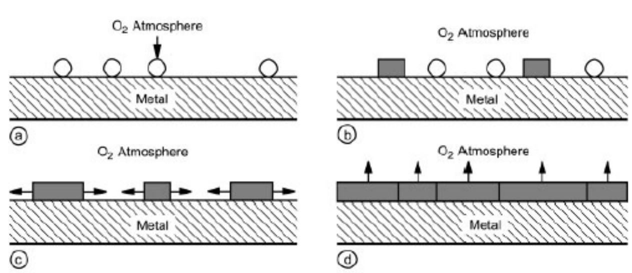


Fig 1 - Phases in the oxide formation on a metal surface a) Oxygen adsorption at the surface, (b) Nuclei formation, (c) Growth of the nuclei in horizontal direction, and (d) Growth of the compact oxide scale in vertical direction [2,4]

Oxide film composition of the alloys differs from that of a pure metal and it is influenced by the type, quantity and number of alloying elements [1,2].

Generally, the oxide layer exists in two forms (Refer to Fig 2) as iron oxides (outer layer) and chromium oxides (inner layer near to the substrate) [1 - 4].

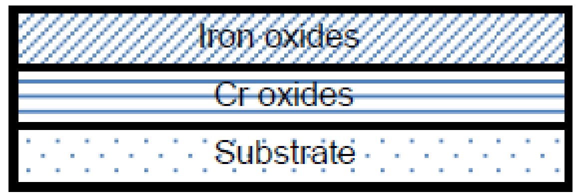


Fig 2 – Oxide composition of stainless steel

Chromium being the main alloying element, forms Cr2O3 and offers oxidation resistance at moderately high temperature. Beyond the scaling temperature (See Table 1), the oxide growth is extremely high due to the large rate of diffusion of oxygen, resulting in a thick oxide layer [1, 2, 5]. This thick oxide layer is loosely attached to the substrate.

Table 1 - Scaling temperature for AMS 5504 (AISI 410) & AMS 5524 (AISI 316) [12]

Steel grade	Composition (%)					Scaling temperature (°C)
	C	Cr	Ni	Mo	N	
AISI 410	0.08	13	-	-	-	830
AISI 316	0.04	17	12	2.7	0.06	830

XRD spectra results on the oxide layer of AMS 5524 by Guillamet et al. shows that the oxide film consists of Fe2O3, Cr2O3 and a spinel oxide (oxide formed by the combination of alloying elements like Mn, Ni, Cr & Fe) [1,6]. According to Montemor, increasing the oxidation temperature in a furnace from 250°C to 450°C, thickens only the external iron oxide while thickness of the internal Cr oxide remains the same for AMS 5524 [1,4].

Cheng et al found that oxide film composition of AMS 5504 varies depending on the oxidation atmosphere. For dry and moist air at 850°C in a furnace, a duplex structure composed of Fe2O3 and Cr oxides were identified, whereas for N_2, a multi layered oxide structure with Fe3O4, $(Fe,Cr)_3O_4$ & little

Fe2O3 were detected. Metallic Fe and $(Fe,Cr)_3O_4$ were the main composition of the oxide layer exposed to 10% $H_2 + N_2$ [1,8].

Of all the cleaning or contaminant removal methods, Laser ablation is considered to be a potential technique for removing oxides from the metal alloys, as it has the capability to remove strongly adhering contaminants and does not leave any secondary residue to dispose other than the contaminants. Researchers have also demonstrated the ability of laser to clean the oxide layer from stainless steel.

In this paper, Nd:YAG laser was used for removing oxide from the stainless steel alloys (AMS 5504 & AMS 5524). Characterization of the laser cleaned area will be performed using SEM and EDS. The objectives of this study are as follows: 1) To demonstrate the ability of Nd:YAG laser to remove oxide from AMS 5504 and AMS 5524 SS alloys. 2) To determine the influence of laser on the cleaned or irradiated surface. This is undertaken because the surface finish is a critical factor in the aerospace component's functionality [9].

2. EXPERIMENTAL METHODOLOGY

A. *Material specifications and heat treatment conditions*

The materials chosen for the laser cleaning trials include AMS 5504 and AMS 5524 sheets of size 120 mm X 60 mm X 2 mm. Oxide layer was formed on the samples by subjecting it to heat treatment (See Table 2).

Table 2 - Heat treatment conditions of all the samples for oxide formation

Alloy	Annealing temperature (°C)	Holding Time (Minutes)
AMS 5504	760	30
AMS 5524	1100	30

The oxide covered sample obtained after heat treatment is denoted as the 'oxidised sample' in this paper.

B. *Experimental setup*

Nd:YAG laser (ROFIN-BAASEL POWERLINE 100D, maximum power of 100 Watts), basically a marking laser was used to remove the oxide from the AMS 5504 and AMS 5524 samples (See Fig 3). After the laser cleaning or removal of oxide from the SS alloys using laser, characterization study on the laser cleaned area was done by scanning electron microscope (SEM), energy and dispersive x-ray spectroscopy (EDS). Images from the SEM (Model: JEOL JSM-5600LV was used to study the microstructural surface characteristics of the cleaned areas.

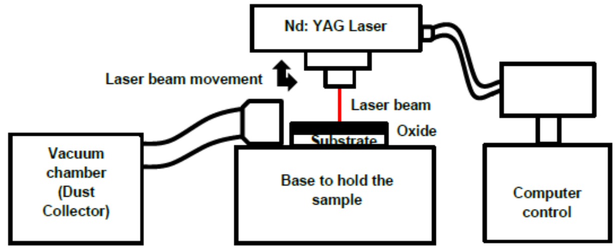


Fig 3 Schematic of the laser cleaning setup

EDS coupled with the SEM was utilized to determine the elemental composition of the surface before and after cleaning.

3. PRELIMINARY EXPERIMENTS

The objective of the preliminary tests was to identify the influence of parameters on the removal of oxide from the SS alloys.

Two trials were undertaken to decide on the parameters for the final tests. At a pulse current value of 36 Amperes, the laser operates at its maximum power of 100 Watts. Based on the SEM and EDS results, the range of parameters will be selected for the final set of experiments.

A. *First trial*

For the first trial, scanning velocity was set to 1000 mm/s and the surface is raster scanned twice. Other parameters employed are given in the Table 3 below:

Table 3 - Pulse current and frequency for the first trial

Parameters	AMS 5504	AMS 5524
Pulse frequency	100 KHz	70KHz
Pulse current	32A and 36A	32A and 36A

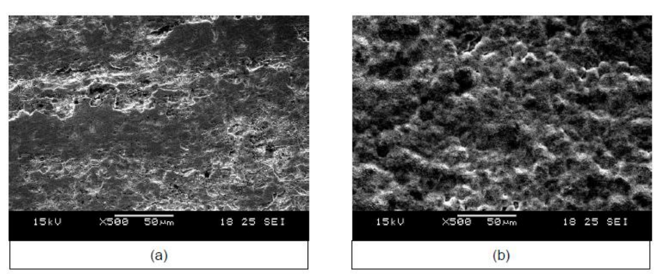


Fig 4 SEM images of AMS 5504 a) Substrate b) Oxide layer

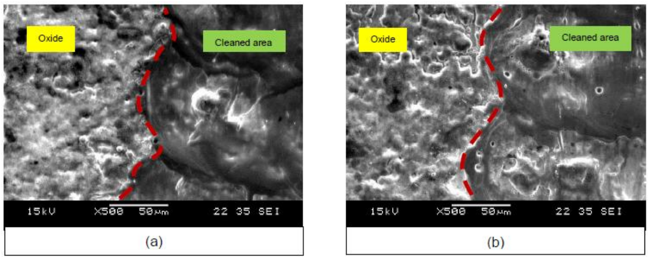


Fig 5 - SEM image of the AMS 5504 laser cleaned area (a) 32A and (b) 36A at a pulse frequency of 100 KHz

Table 4 - Elemental composition of AMS 5504 from the EDS

AMS 5504		Carbon	Oxygen
		Weight (%)	
Oxidized sample		3.2	29.32
Substrate		6.02	Nil
Pulse frequency 100 KHz	32A	3.39	26.45
	36A	4.00	25.76

From the SEM images (Fig Fig 5), it is clear that laser cleaning has altered the surface characteristics. From the EDS results (Table 4), oxide is removed as indicated by the decrease in percentage of oxygen from that of the oxidised sample. It is observed that oxide removal increases with the pulse current. Also, carbon % increases with the pulse current. Similar results were obtained for AMS 5524 (Table 5).

Table 5 - Elemental composition of AMS 5524 from the EDS

AMS 5524		Carbon	Oxygen
		Weight (%)	
Oxidized sample		4.22	28.34
Substrate		8.77	Nil
Pulse frequency 100 KHz	32A	3.71	22.49
	36A	3.35	22.06

B. *Second trial*

AMS 5504 & AMS 5524 SS alloys were raster scanned with the scanning velocity of 500 m/s and the number of passes was increased from 2 to 5 compared to the first trial. When the second trial samples were viewed under optical microscope, surface coloration was observed which indicates only partial removal. Findings from the preliminary experiments show that oxide can be removed from the surface by laser ablation.

4. PARAMETERS FOR THE FINAL SET OF EXPERIMENTS

Based on the preliminary results, the parameters for the final set of experiments were fixed.

Table 5 – Parameters for the final set of experiments

Parameters	
Scanning velocity	500 mm/s
Number of passes	6
Pulse frequency	50KHz
Pulse current	36A

5. FINAL SET OF EXPERIMENTS

In this section, SEM images, and elemental composition from the EDS for the substrate, oxidised sample and laser cleaned areas for the final set of experiments are presented. As previously mentioned, cleaning of the oxide from AMS 5504 & AMS 5524 substrate were achieved by raster scanning the surface six times (i.e. 6 number of passes) with Nd:YAG laser at 36A pulse current and various pulse frequencies (See Table 5). The scanning velocity was kept constant at 500 mm/s. The dimension of the cleaned area for each pulse current and frequency was set to 11mm x 11mm with the help of a computer. Cleaning is carried out only in the upper half part of the samples as shown in Fig 6.

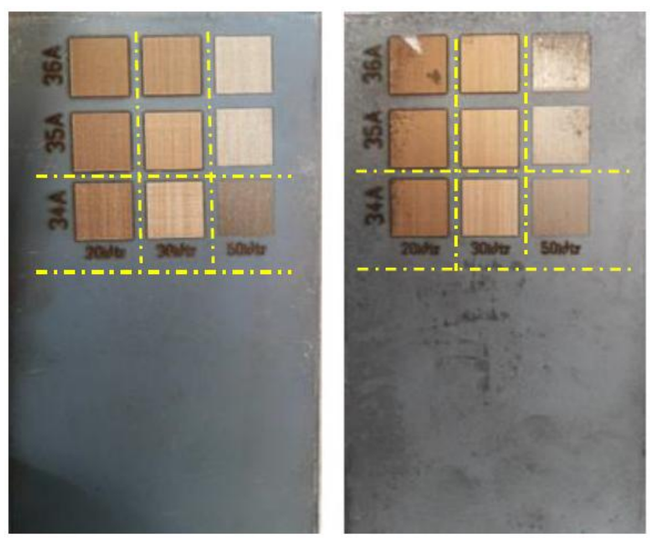


Fig 6 AMS 5504 and AMS 5524 samples before and after cleaning (left to right)

6. EXPERIMENTAL RESULTS

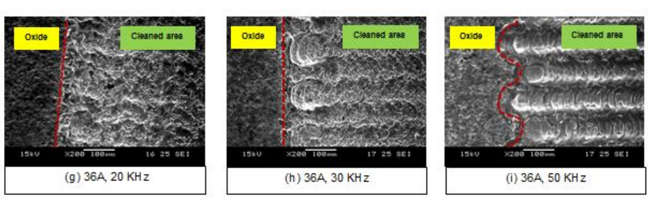
A. Equipment setup

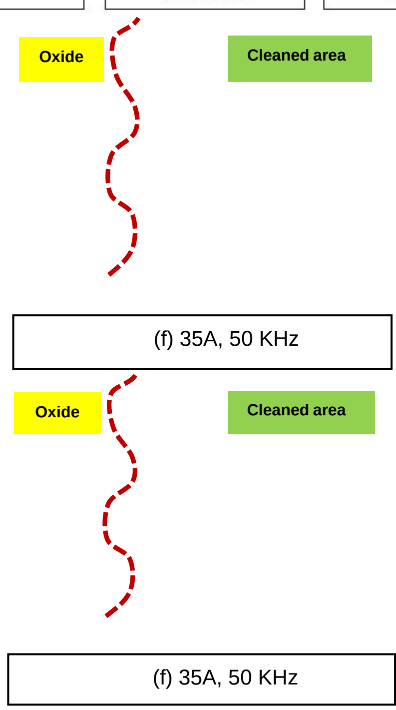
Since the SEM operates in vacuum, sample should be placed within the base of the closed chamber to obtain the images and elemental composition from EDS. It is because of this reason; all the laser cleaned samples were cut into small pieces with the shear cutter. The yellow dash-dot lines indicate how the samples were cut for the SEM and EDS analysis (Fig 6).

B. SEM and EDS analysis of AMS 5504

The accelerating voltage in the SEM was set to 15 KeV and all the images of the laser cleaned areas were of magnification 200x.The spot size was changed from 25 to 35 and the magnification of the image was reduced from 200x to 50x in order to cover a large area for the EDS analysis.

From the SEM images, it is clear that laser cleaning has altered the surface characteristics (Fig 7).





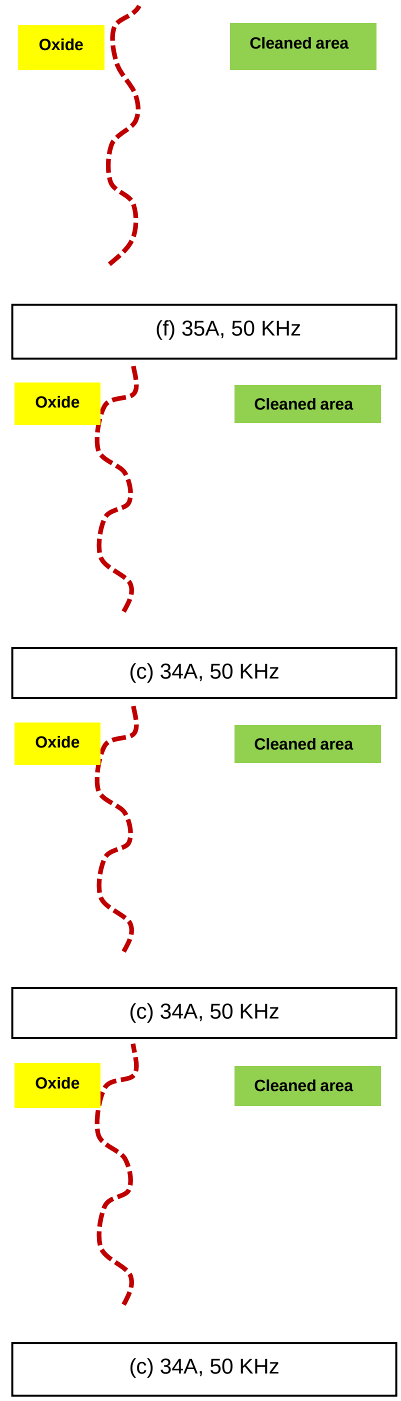


Fig 7 SEM images of the AMS 5504 laser cleaned areas (36A, 50KHz)

Surface melting has occurred for all the pulse frequencies employed, leading to the formation of re-cast layer which will have different mechanical and metallurgical properties to that of the substrate (Fig 4 (a)) and oxidised sample (Fig 4 (b)). The laser irradiated area is suddenly cooled at room temperature after the cleaning operation; melted layers re-solidify to form the re-cast layers.

Table 6 Elemental composition of carbon & oxygen from the EDS

AMS 5504		Carbon	Oxygen
		Weight (%)	
Oxidised sample		3.2	29.32
Substrate		6.02	Nil
Pulse current 36A (100Watts)	20KHz	15.05	12.08
	30KHz	12.45	9.07
	50KHz	7.95	7.7

Since the experiments were performed in ambient air, intense localized heating of the surface by laser might have led to increase in the carbon content (See Table 6). Most of the laser cleaned areas appears dull with brown coloration instead of the bright shiny metal surface (See Fig 6). For all the frequencies employed, oxide is significantly removed from the substrate, as could be seen from the reduction in oxygen % to one-third to that of the oxidised sample (See Table 6).

C. SEM and EDS analysis of AMS 5524

The laser cleaned areas of AMS 5524, should originally appear brighter like a shiny metal, instead a brown coloration was observed (See Fig 6 second from the left). The reason may be carbonization of the sample as the cleaning trial was conducted in ambient air.

Behaviour of the AMS 5524 laser cleaned areas were similar to that of AMS 5504 with the formation of re-cast layer due to surface melting as shown in Fig

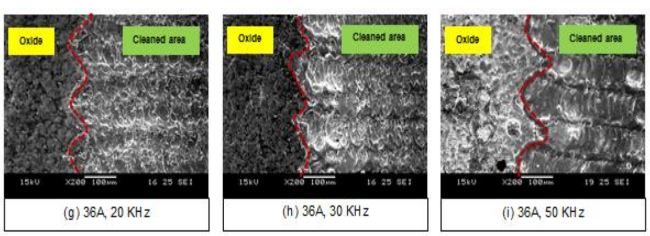


Fig 8 SEM images of the AMS 5504 laser cleaned areas (36A)

Since melt patterns of the AMS 5524 were similar to that of AMS 5504, it can be concluded that ablation is the mechanism responsible for removal of oxide from AMS 5524 substrate. Elemental composition of the AMS 5524 oxidised sample, substrate and laser irradiated surfaces were shown in Table 7.

Table 7 - Elemental composition of AMS 5524 from the EDS

AMS 5524		Carbon	Oxygen
		Weight (%)	
Oxidised sample		4.22	28.34
Substrate		8.77	Nil
Pulse current 36A	20KHz	12.78	12.10
	30KHz	10.86	8.93
	50KHz	6.26	12.68

Significant decrease in oxygen % in the laser irradiated areas (See Table 7) shows that oxide is removed from the surface. But the removal is not complete with some amount of oxygen still present in the cleaned areas.

7. CONCLUSION

The main objective of this paper was to study the influence of Nd:YAG laser on the removal of oxide from the stainless steel alloys (AMS 5504 & AMS 5524).

Results from the preliminary experiments helped in selecting the parameters for final set of experiments. Characterization of the laser cleaned areas was carried out with techniques such as SEM and EDS. The following were the findings from final set of experiments:

- As a result of laser irradiation, surface melting has occurred and re-cast layers were found in SEM images of all the laser cleaned areas of AMS 5504 and AMS 5524. These re-cast layers are formed on the metal surface due to intense localized heating of the laser beam and sudden cooling at room temperature after the laser irradiation.

- It is evident from the melt patterns (in the form of ripples) of AMS 5504 and AMS 5524 that oxide is removed from the surface by vaporization and melting. So it is concluded that ablation is the mechanism responsible for removal of oxide and scale from the metal alloys.

- Significant reduction in oxygen % for all the pulse currents and frequencies employed shows the ability of Nd:YAG laser to remove oxide from the stainless steel alloys (AMS 5504 & AMS 5524).

- Freshly formed oxide is suspected to be present along with the re-cast layers as considerable amount of oxygen % is still present in the laser cleaned areas of AMS 5504 and AMS 5524.

8. FUTURE SCOPES

In near future, instead of pulse current, laser ablation will be performed based on the laser fluence. Experiments will be done with the samples placed inside the closed chamber filled with the inert gas to avoid oxide formation during laser ablation. His area of interests includes laser ablation, material characterization, bio-composites and mechanical testing.

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