

# **MICROSTRUCTUR AND MICROHARDNESS OF LASER CLADDING Ni BASED ON COLD ROLLED STEEL**

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## **ABSTRACT**

 A study is reported of the laser cladding of a nominal composition of Ni 5 wt% Al on cold rolled low carbon steel (0.16 wt% C), using a high power continuous  $CO<sub>2</sub>$  laser. The severe rolled microstructure of steel was changed considerably at the heat affected zones under all specific energies. The cladded coatings showed the presence of  $\gamma$  solid solution and  $\beta$ (NiAlFe) phases. Sound metallurgical bonding with absence of porosity and cracks was observed between the substrate and the clad coat at specific energy higher than 80 J/mm2. The ferrite and pearlite microstructure of the substrate was changed to martensite at the region adjacent to the clad interface. It followed by large grains of austenite/ferrite and pearlite (grain growth zone), fine grains of austenite/ferrite and pearlite (recrystallization zone) and very small zone of relatively small change of cold structure (recovery zone). The last zone was confirmed by micro hardness as a recovery zone.

 This investigation confirms clearly the possibility of formation different structures of grain growth, recrystallization and recovery at the laser heat affected cold rolled low carbon steel. The observed results suggest the developing of a new technique to obtain tentative functionally graded material.

**KEYWORDS : Cold rolled steel; High power laser; Functionally graded material; Recrystallization; Grain growth; Recovery**



### **الخالصة**

 تضمن البحث دراسة االكساء بالليزر نو القدرة العالية نوع ثاني اوكسيد الكربون للصلب الكربوني المدرفل على البارد والحاوي على %wt 0.16 كربون باستخدام مزيج من مسحوق النيكل الحاوي على %wt 5 المنيوم. تغييرت البنية المجهرية المدرفلة بشدة للصلب بصورة مستمرة في المنطقة المتأثرة بالحرارة عند كل قيم الطاقة النوعية المستخدمة. اوضحت طبقات الاكساء الناتجة وجود طور المحلول الجامد كاما و بيتا (المركب الوسطي من النيكل والألمنيوم والحديد). تم الحصول على ربط ميتالورجي خالي من العيوب عند الحد الفاصل بين طبقات االكساء وسطح الصلب عند قيم طاقة نوعية اكبر من 80 جول/ملم<sup>2</sup>. تـحولت البنية المجهرية للفرايت والبيرلايت في الصلب الى المارتنزايت عند الحد الفاصل للاكساء بعدها تكونت البنية المجهرية من حبيبات من الاوستنايت / الفرايت والبير لايت ذات حجم كبير (منطقة النمو البلوري) وبعدها حبيبات ناعمة من الاوستنايت ولفرايت والبيرلايت (منطقة اعادة التبلور) واخيراً منطقة صغيرة جدا ذات بنية مجهرية تختلف قليلا عن البنية المجهرية للصلب المشكل (منطقة الاستعادة) . أو ضحت النتائج الحصول على تطوير تقنية جديدة لتكوين مواد متدرجة وظيفيا كاذبة .

#### **INTRODUCTION**

 The Ni-Al alloys contain high amount of Ni have been considered for long time as important alloys having high mechanical properties to be used at high temperatures Darolia et al. (1996). The Ni rich alloys with Al is consisting of high volume fraction of intermetallic compound B2 phase of NiAl (β) with L12 ordered phase of y' (Ni3Al) in the matrix of Ni-solid solution (y) Chenf et al. (1998). On the other hand, the rapid solidification melting or coatings techniques may play an important role to enhance further the properties of Ni-Al alloys Wei et al. (1993), Zhang et al. 2011). The rapid solidification of Ni-Al alloys could be achieved by laser cladding to produce rapid (solidification deposits on many cheap substrates such as plan carbon steels Dutta Majumdar and Manna (2011), Toyserkani et al. (2005). Many advanced technologies are getting great benefit from laser surface engineering. Among of these is laser cladding which have high flexibility to deposit many alloys. It has many advantages compared with many surface coating techniques Jayakumar et al. (2015), Torims (2013), Birger et al. (2011). It characterizes with small control dilution region, rapid solidification, clean surface and narrow heat affected zone Komvopoulos and Nagarathnam (1990), Tibblin (2015). Laser cladding using continuous feeding of a powder into the laser melt pool has the most powerful coating technique to deposit many different alloys on substrates. These cladding are capable for melting control depth of substrate and the feeding powder. The clad coatings have excellent properties of homogenous chemical composition, novel microstructure, sound interface, high hardness, excellent wear and corrosion resistance Zhong and Liu (2010), Soodi (2013), Xu et al. (2017). These features of clads may have extra combined improvement of the substrate due to the phase transformation associated with heat transfers from the fusion of clad region down to the substrate Barekat et al. (2016), Mahmoud and El-Labban (2014). The most important structural alloys widely used for many applications which require medium mechanical properties and chemical stability are low carbon steels Lin et al. (2013). These materials may consider as important cheap substrates for deposition clad coatings for many high corrosion and oxidation resistance alloys such as Ni-Al alloys Li et al. (2006). At the same time to modify the microstructure of cold rolled low carbon steel to maintain high ductility with medium strength, the acceptable and applicable strengthening mechanism is the grain refining by recrystallization Kanga et al. ( 2010). Generally, laser heating or melting of steels could increase the hardness due to the local phase transformation Dutta Majaumdar and Manna (2003), Kempena et al. (2011), Lee et al. (2009). This is only correct for normalized microstructure. Presence of severe deformed microstructure may produce complex responses. It appears that the laser cladding of cold rolled low carbon steel with Ni-Al alloy can enhance two important properties. These are wear and corrosion resistance from the clad deposit and high ductility and strength from the heat affected zone.

### **EXPERIMENTAL PROCEDURES**

Samples of cold rolled low carbon steel with 0.16 wt% C (Fe-0.16% C, 0.6-0.9% Mn,  $\leq$ 0.05% S and  $\leq$  0.04% P) with dimensions 5 cm x 2 cm x 0.3 cm were used as substrates for laser cladding. Laser cladding of the shot blasted substrate were carried out using a high power continuous wave  $CO_2$  laser. The laser power (P) employed was 1.8 kW. The samples were traversed relative to the defocused 2.5 mm laser spot size (d) at different speeds (V) (1.5, 3.2, 4.8, 6.6, 9.1 and 12.5 mm/s). The corresponding specific energies (P/dV) are 480, 225, 150, 109, 79 and 58 J/mm<sup>2</sup> . The average particle sizes of nickel and aluminum powders are 55 and 63  $\mu$ m respectively. The feeding rate of the injection premixed powder of Ni- 5 w%t Al was 10 g/min. Low rate of argon gas (5 SLPM) was blown to protect the melted pool from oxidation. Table 1 listed the laser parameters and laser processing parameters employed.

 The processed laser clads were examined metallographically using standard metallography procedures for optical and scanning electron microscopy. The samples were cut perpendicular to the cladding thickness using wire cutting. The cutting samples were cold mounted and then grinding with 500 and 1000 SiC grid emery papers. These ground samples were polished with 1 µm diamond paste. Two etching solutions were used, 3% Nital to resolve the microstructure of heat affected zones and substrate and a mixture of 50% cm<sup>3</sup> HCl and 50% cm<sup>3</sup> HNO<sub>3</sub> for clad regions. The microhardness of the transverse clad region and different regions of heat affected zones and substrate were measured from Vickers microhardness test using 300 g load. The microhardness of the different zones was measured by determining the corresponding microstructure from the microscopy equipped with microhardness tester.

 The chemical compositions of the clad regions were measured using energy dispersive spectroscopy (EDS) equipped with scanning electron microscopy. More detail regarding the experiment work can be found elsewhere Maryam et al. (2017).

#### **RESULTS AND DISCUSSION**

Figure(1) shows the microstructure of the cold rolled low carbon steel at different magnifications. It is clearly reveals the heavily deformed grains of perlite and ferrite in the direction of the deformed occur. Higher magnification of the microstructure shows the blocks of the phases which highly raise the texture effect. This microstructure with dependent direction mechanical properties has a detrimental effect. Therefore, it is very necessary to overcome this disadvantage by heat treatment. Figure(2) shows the typical low magnification of cladded samples in which the laser performs the formation of clad due to interaction between the incident laser beam with the powder. Melting of the powder as well as part of the substrate was occurred immediately at a time dependent on the traverse speed and laser beam diameter  $(t = d/v)$ . Then the melt pool was advanced further through the substrate where the solidification taken place by rapid heat conduction from the substrate. These conductions of solidification produce metallurgical bonding without any defects of porosity and cracks as can be shown in figures (3 and 4). As a result of increasing the specific energy the melting of the substrate and the dilution were increased. Higher specific energy has two adverse typical effects; firstly, increasing the thickness of the clad coating (Table 2) and area of heat affected zone (Figure 5). Secondly, the chemical composition of clad region changes continuously from the chemical composition of the injected powder from the upper surface of clad toward the interface (Figure 6). This behavior is related directly to the dilution of the clad region from the steel substrate. There are wide spectrum of thickness and composition of the clad coatings at different specific energies. In general, the minimum dilution with a sharp metallurgically bonded interface should be obtained to avoid the change in chemical composition from the starting powder. The interesting features were observed from the adjacent phase transformation and heat affected zones below the bonding interface. These features are totally different than those obtained from laser cladding on hot rolled substrates Ellis et al. (1995). Figure (6) shows higher magnification of the typical microstructural changes of the deformed microstructure of the substrate to produce different microstructure associated with the heating the substrate. Figures( 2 and 6) postulate clearly the duplex mechanisms of martensitic phase transformation and recrystallization. There are four zones formed below the bonding interface namely (i) martensitic phase transformation, (ii) grain growth, (iii) recrystallization and (iv) recovery. The formation of martensitic zone (Figures 6d and e) is due to rapid cooling from the austenitic formation range. The temperature is believed to be higher than 1000  $\rm{^{\circ}C}$  (according to the Fe-C phase diagram). The presence of some grains of austenite (not ferrite) is due to the lower diffusion time which is not sufficient to produce homogenous super saturated of carbon in austenite (Figure 6c). This was confirmed from microstructure due to the absence of equiaxed and interconnected grains associated with ferrite.

Therefore, the microstructure is consisted of small amount of austenite, ferrite and perlite. The low temperatures rises at the substrate below the martensitic zone suggest that the temperatures are less than 1000  $\degree$ C and may reaching less than 300  $\degree$ C near to the substrate (Figure 6b). The short interaction times which are sufficient to all these heat treatments of grain growth, recrystallization and recovery are believed to be due to the high stored internal energy in the deformed substrate. Higher temperatures below to the temperature of martensitic transformation are sufficient to produce grain growth of the deformed microstructure (Figures 6c and d) .

 Microhardness measurements for all zones below the clad coatings showed values higher and lower than those of the deformed substrate. The maximum hardness was found at the martensitic phase transformation zone and the lower at the grain growth zone (Table2). The higher hardness of the heat affected zone was observed at the martensitic transformation zone while the lower hardness was found at the grain growth zone (Figuress. 3, 4 and 6 and Table 2). The lower hardness of the grain growth zone is due to complete removal of line defect (dislocation) and point defect present in the deformed structure. Careful examination of the deformed structure and the formation of austenite structure at room temperature suggest the heavy deformation and high stored energy. This is evident from the lower interaction time which is sufficient for changing the structure due to the stored energy. It should be reported that the hot rolled substrate will produce totally different heat affected zones Ellis et al. (1995). Optical microscopy and scanning electron microscopy analysis of the transverse sections of the single clads show that the chemical composition of the clad regions depends heavily on specific energy. The chemical compositions of the clad regions are shifted from the standard mixed powder. The average amount of Ni at the clad regions is far from the standard value at the mixed powder as observed from EDS and EPMA (Figure 7). This is explained previously due to the strongly dependent on the area dilution associated with processing parameters Maryam et al. (2017). The laser surface cladding of low carbon steel lead to laser melting of part of the substrate which mixed with melted powder into the melt pool producing clads with different dilution from the substrate .

 Examination of the microstructure shows the less dependent of the microstructure on laser processing parameters. This was found to be due to the phases formed which are mostly based on the Ni solid solution contains different amount of iron and aluminum (Figure 8). The phases produced are mostly Ni solid solution  $(y)/Ni_3A1(y')$  and intermetallic compound based on NiAlFe  $(\beta)$ . The amount of y phase observed is small compared with y'. The microstructures of the clad regions are cellular structure due to the rapid cooling rates. These high cooling rates are due to the rapid solidification which generates heating to the adjacent substrate with different cooling rates dependent to the distance from the cladding interface (Figures. 2, 4 and 7) .

### **CONCLUSIONS**

1- Thin single tracks with typically 80 to 580 µm clad thickness was produced without defects. The hardness of clads varies between 1.90 to 2.8 GPa at the outer surfaces dependent on the specific energy.

2- The microstructure of the laser cladding of Ni-5 wt% Al do not depend on the laser specific energy regardless the different percentage of dilution from the steel substrate. They consist of Ni- solid solution ( $\gamma$ )/ $\gamma'$  (Ni<sub>3</sub>Al) and β (NiAl) phases.

3- Four different heat affected zones namely martensitic, grain growth, recrystallization and recovery were formed. The maximum hardness was found at the martensitic region near the interface between clad and heat affected zones.

4- The deformed ferrite and pearlite microstructure of cold rolled low carbon steel substrate changed to austenite/ferrite and pearlite at the center of the heat affected zone.

TEM laser mode	00
Raw laser beam diameter, D	$20 \text{ mm}$
Focal length, f	$125 \text{ mm}$
Shrouding gas, Argon	2.2 SLPM
Laser power, P	$1.8$ kW
Laser beam diameter, d	$2.5 \text{ mm}$
Traverse speed, V	$1.5 - 12.5$ mm/s
Interaction time $(d/V)$ , t	$0.2 - 1.67$ s
Power density, $P_A$ (4P/ $\pi d^2$ )	$367$ W/mm <sup>2</sup>
Specific energy, S (P/dV)	58-480 J/mm <sup>2</sup>

**Table 1.** Laser and processing parameters studied.

**Table 2.** Typical Microhardness measurements at different regions of clad and heat affected zones for different specific energies.

Specific energy			480 J/mm <sup>2</sup> 150 J/mm <sup>2</sup> 109 J/mm <sup>2</sup>	79 J/mm <sup>2</sup>	
Location					
Total thickness of clad region, $\mu$ m	579	256	146	78	
50 µm from outer surface	282	263	218	188	
Centre of the clad region	266	252	212		
50 µm from the interface	216	186	180		
Martensitic zone	412	422	418	421	
Grain growth zone	124	118	122	118	
Recrystallization zone	196	201	185	193	
Recovery zone	245	242	252	246	
Cold rolled substrate	260	260	260	260	



**Fig.1.** Microstructure of cold rolled low carbon steel (a) optical microscopy, (b) and (c) scanning electron microscopy showing the deformed ferrite and perlite at the rolling direction.



**Fig. 2.** Typical transverse section views of Ni-5 wt% Al clad at specific energies showing the clad regions and different zones formed at the heat affected zone (a) 220 J/mm<sup>2</sup>, (b) 150 J/mm<sup>2</sup> and (c) 48 J/mm<sup>2</sup>.



- **Fig. 3.** Typical scanning electron micrographs of transverse section of Ni-5 wt% Al clad (a) low magnification showing the sound clad and interface,
	- (b) low magnification of interface clad coating, interface, different regions of heat affected zone and substrate and
	- (c) higher magnification showing the martensitic structure near the interface.



**Fig. 4**. Typical optical micrographs of transverse section of Ni-5 wt% Al clad (a) low magnification showing the sound clad and interface, martensitic structure near interface and mixture of equaxied autenite and perlite,

(b) grain growth region of autenite and perlite, fine cells of austenite and perlite at recrystalization region, slightly changed of deformed structure at recovery region and rolled structure at the substre and

(c) Higher magnification showing the recrystalization and recovery zones.



 **Fig. 5.** The average wt% of Ni, Al and Fe in the center of clad regions processed at different specific energies.



**Fig. 6.** Scanning electron micrographs showing the detail of clad region and heat affected zones **(a)** low magnification,

- **(b)** the functionally graded structure of recrystallization, recovery and substrate
- **(c)** interface between grain growth and recrystallization zones
- **(d)** zones of martensite and grain growth zones and
- **(e)** detail of martensite and grain growth zones.



**Fig. 7.** EDS analysis of clad region processed at 100 J/mm<sup>2</sup> specific energy **(a)** near interface and **(b)** at the center of clad region.



 **Fig. 8.** Typical microstructure of clad region showing the presence of ɣ/ɣ' and β phases.

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