

## The effect of laser and thermal treatment on the hardness and adhesion force on the ceramic coating by thermal spray technique

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### Abstract

Ceramic coating compose from a ceramic mixture (MgO, Al<sub>2</sub>O<sub>3</sub>) and metall (Al-Ni) were produced by Thermal Spray Technique. The mixed ratio of used materials Al:Ni (50%) and 40% of Al<sub>2</sub>O<sub>3</sub> and 10% MgO. This mixture was spray on a stainless steel substrate of type (316 L) by using thermal spray with flame method and at spraying distances (8, 12, 16 and 20) cm, then the prepared films were treated by laser and thermal treatment. After that performing a hardness and adhesion tests were examined. The present study shows that the best value of the thermal treatment is 1000 °C for 30 mint; the optimum spray distance is 12 cm and most suitable laser is 500 mJ where the microscopic and mechanical characterstic of coating layer get well.

### Key words

Thermal spraying flame, thermal tretment, laser tretment, ceramic materiale.

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## تأثير الليزر والمعالجة الحرارية على الصلابة وقوة الالتصاق على الطلاء السيراميكي بواسطة تقنية الرش الحراري

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### الخلاصة

تم انتاج طبقة طلاء سيرميكية مكونة من خليط سيراميكي (MgO، Al<sub>2</sub>O<sub>3</sub>) ومعدني (Al-Ni) حيث كانت نسبة المادة الرابطة (Al:Ni) هي ثابتة (50%) وكانت نسبة Al<sub>2</sub>O<sub>3</sub> (40%) بينما MgO كانت (10%)، تم رش الخليط على ارضيات من الفولاذ نوع (316 L) باستخدام جهاز الرش الحراري باللهب ولمسافات رش مختلفة (8، 12، 16، 20) سم، اجريت المعاملات الحرارية والليزرية لطبقة الطلاء المتكونة واخذت الصور المجهرية لها، بعد ذلك تم اجراء فحوص الصلادة وقوة الالتصاق بعد المعاملات المطلوبة. اظهرت نتائج الفحص ان افضل درجة معاملة حرارية كانت (1000 °C) ولزمن ساعة ونصف، وافضل مسافة رش 12 سم، واحسن طاقة ليزرية (500 mJ)، اذ تحسنت خواص طبقة الطلاء الميكانيكية والمجهرية.

### Introduction

In the last years, a great effort paid for employing ceramic-metallic coating layer in wide fields such as electrical [1] and thermal applications [2], which provides a good isolation features over a wide high-temperature ranges (thermal barrier coating)

especial jet plan engines, marine engines and gaseous turbines [3].

Ceramic coatings provide high-performance oxide layers on metals and alloys as wear resistance to protect against corrosion or friction. Also it can be used as heat or electrical insulation. Some ceramic coatings can

be prepared by different techniques such as spray coating [4], laser deposition [5], sputtering coating [6], Sol – gel method [7] and cathodic arc plasma deposition [8].

The composite materials usually affected by the materials made from, which included the base (matrix) and reinforcing phase. The base usually represented by the continuous phase in the composite material, which binding all elements and reinforcing material, as well as connecting the parts together in order to form a coherent structure that can produce excellent mechanical properties. Such that increasing the strength and lighten in weight. So that more attention to the production of the composite material to be a suitable alternative for the tradition engineering material like metals, alloys, and polymers. The reinforcing material function is to strengthen the base material which may be ceramic, or metallic or polymers of different forms, such as powder or fibers or peels[9]. The production of composite material in last years has been developed incredibly especially material reinforced with fibers or particles or bristles, especially that are used in casting, powders technology or thermal spray coating [9,10]. Thermal spray is utmost methods among the spray techniques [11] which can be used with a wide range of material ranged [12] from the low melting points material to the high melting point material and the composite material [13]. It is used to obtain a physical and mechanical acceptable characteristic for the coating process. These techniques consider as a most important industrial method [14] for the superficial example required for coating big parts with high sedimentation efficiency. These Techniques required high precision and control of the coating condition such or material feeding, air pumping

and the distance between the spray gun and the base to get high adhesion coats and extra mechanical and structured properties [9].

## Experimental part

### 1- The employed raw material

We have used a binding material made by a Swedish company—castolin+ Eutectic, its function is that production chemical interactions regions make a stable form. The (Al-Ni) alloy has grain size ranged (75-100)  $\mu\text{m}$ , for resistance the oxidation in high temperatures and to the homogeneity of its fusible and adhesion with the base substrate, we also used the  $\text{Al}_2\text{O}_3$  powder made by Metco Company or matrix with grain size range (70-230)  $\mu\text{m}$  with 40 %, which has great isolation properties and wear resistance at high temperatures. For the coating material, we measure the grain size with another method which knows as sieving. These grain sizes are sufficient to melt with thermal, also we used (10 %) MgO with grain size 75  $\mu\text{m}$  (Metco) in order to increase the stability the Alumina phase and Pervert wearing when the phase change.

### 2-Preparation method

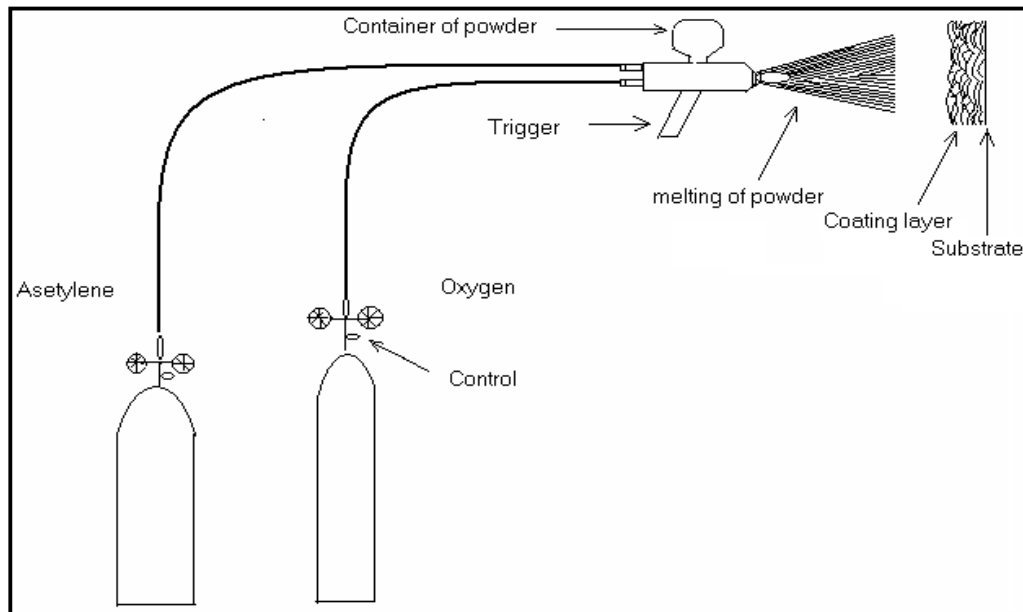
The cermet compound was prepared from 50 % (Ni-Al), 40 % from the base powder of  $\text{Al}_2\text{O}_3$ , and 10 % from MgO in order to stabilize the Alumina phase, after that the powder mixed well by using an electric mixer for 3 hr. To get a homogeny mixture, we apply thermal treatment for the powder of cermet compound before the coating process at 150  $^\circ\text{C}$  for 30 min using an electric oven provided with heat control. The aim of thermal treatment before the coating process is the dryness the powder grain from humidity so that these grain will be in a good lithe state and to be ready for production coating with a sufficient adhesion force. We

depend on adding the binding powder with a ratio of 50% by weight [9] to the substrate. The preparation process the surfaces samples before the thermal spraying very important point for the success the coating process, because the temperature of adhesion the coating material with substrate surface depend, only on the mechanical entanglement of the solidified grain or the substrate surface and after cutting and softening the samples to the required dimensions to place on the samples holder, then these samples washed with alcohol for removing fact and avoid touch it during the preparation process we had used Grit Blast instrument (homemade) for the sake of increasing the harshness the sample surfaces by employing the sand grain ( $\text{SiO}_2$ ) with diameters (0.7-1.5) mm. After the harshness process finished the sample re-cleaned. The preparing and coating processes must be very fast. The most important variable parameters of spraying is to select the coating type and the distance between the bases spray Gun spray angle, the harshness of the bases and the thickness of coating layer. Table 1 shows the most important features of spraying process parameters we had to obtain in the experimental tests and finally the

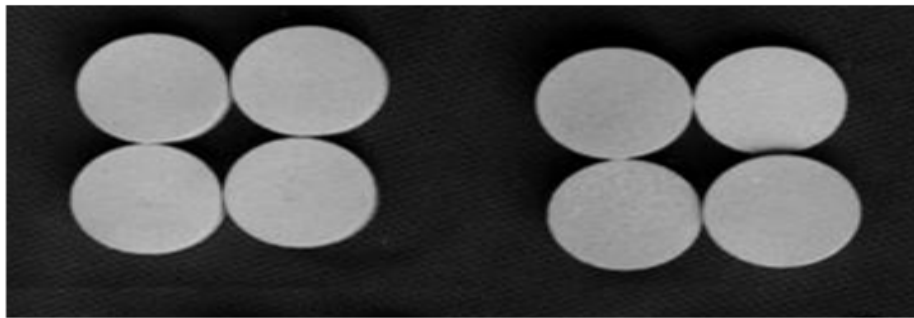
coating process applied to the prepared samples with thermal spraying methods by flame. We use this method in coating all samples under study Fig.1 illustrates the system of spraying by flame. By burning the gas fuel, we obtain the melting temperature of coating powder, which runs by controlling Estelline and oxygen mixture. Where the best values was a 1 bar oxygen and 0.7 bar for Estelline. Through a special material, are fed through crucible placed on the upper part of the system and controlling the open and close the value. Through the spraying process, when these powders reach the region of gases flame, it will be molten either partially or totally, while the applied pressure will transfer the drops of the molten quickly toward the substrate and thus burned gases pressure transfer the molten drops up to velocity  $100 \text{ m.s}^{-1}$ . Fig.2 shows the samples prepared by spraying methods by flame. The spraying process at different distances to obtain the optimum spraying distance followed by this systems. The spraying distances are 8, 12, 16, 20 cm which represents the spray parameter with spraying angle  $90^\circ$ .

**Table 1: The thermal spraying parameters by flame for preparing the cermet compound ( $\text{Ni-Al+Al}_2\text{O}_3+\text{MgO}$ ).**

OXY – Acetylene Mixing	4 / 0.7
Spraying Distances	(8,12,16,20) cm
Thickness Coating	$1.35 \pm 0.3 \text{ mm}$
Flame spray temp.	$\approx (3000)^\circ\text{C}$



*Fig.1: Schematic of system for spraying by flame.*



*Fig.2: Samples prepared by flame spraying method.*

### 3-Test and experimental measurement

#### 1-Optical microscope test

Here, we test the samples microscopically which had been coated in order to observe its fine structure at spraying distance (8, 12, 16, 20) cm. After that, we test samples microscopically after thermal and laser treatments. For the microscopic structure of the sample were prepared very well which associated with softening and polishing processes.

#### 2-Heat treatment

The thermal treatment (sintering) on the coating samples for the composite compound to get a ride from the porosity which weakens the layer

coating features. The thermal treatment ambiances normal condition by using an electric oven and these samples placed at temperatures 1000 °C and 1150 °C for go min, in order to notes the influence of thermal treatment and treatment period on the coating layer features are to get a relatively stable interior structure.

#### 3-Laser treatment

By laser treatment, we executed the treatment through using laser over coating .layers which containing idea features, so or to enhance the physical and mechanical for coating layer after treatment. The laser used to have in (Neodymium- Yag) of maximum energy 500mJ n sample. Treatment

after applying several laser energies (400, 460, 500) mJ for time interval 10 sec at distance 50 cm.

#### 4-Hardness test

The Vickers Hardness had been measured using an Instrument called METKON, and the macroscopic hardness of the samples which coated had been measured then thermally treated, after preparation such as softening and polishing these samples. The employed trace is pyramid sample like, and the user load was constant about 100g in 10sec, then automatically raised after lighting the optical pointer in the required time, then the trace demission had been calculated in two dimensions and; directions for five measurements and taking it average for getting the

hardness value from the digital screen on the instrument directly and the equation represent Vickers are hardness:

$$H_V = 1.8544 \times \frac{p}{(d_{av})^2} \quad (1)$$

where  $H_v$ : is Vickers hardness in  $g/mm^2$ ,  $p$ : the applied load (g) and  $d_{av}$ : the average value of trace diameter.

#### 5-Adhesion test

The adhesion of prepared coating layers was tested by Micro Computer Controlled Electronic Universal Testing Machine (WDW-50E), made by Time Group Inc, according to the standard ASTM A370. The samples were fixed on the adhesion machine as shown in Fig.3.

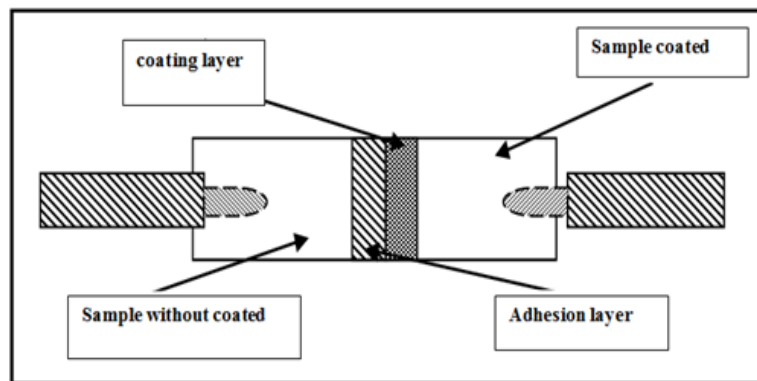


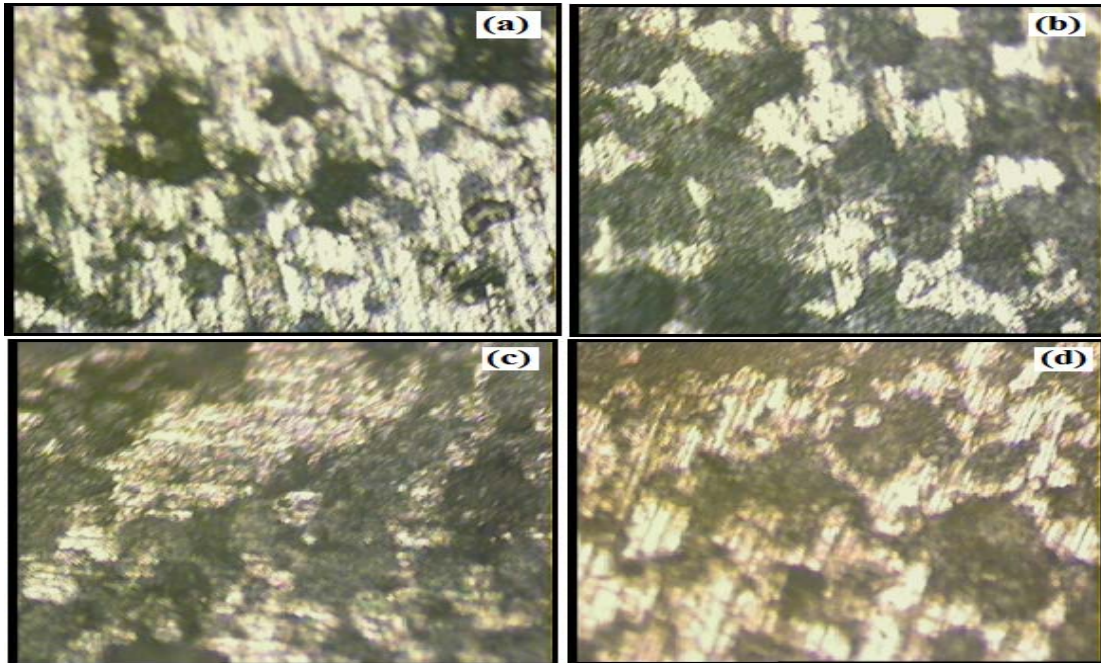
Fig.3: Schematic for samples fixed on the adhesion machine test.

### Results and discussions

#### Microscopic structure of coating layer after thermal treatment.

Fig. 4 (a, b, c and d) shows the effect of heat treatment applied at temperatures of 1000 and 1150 for

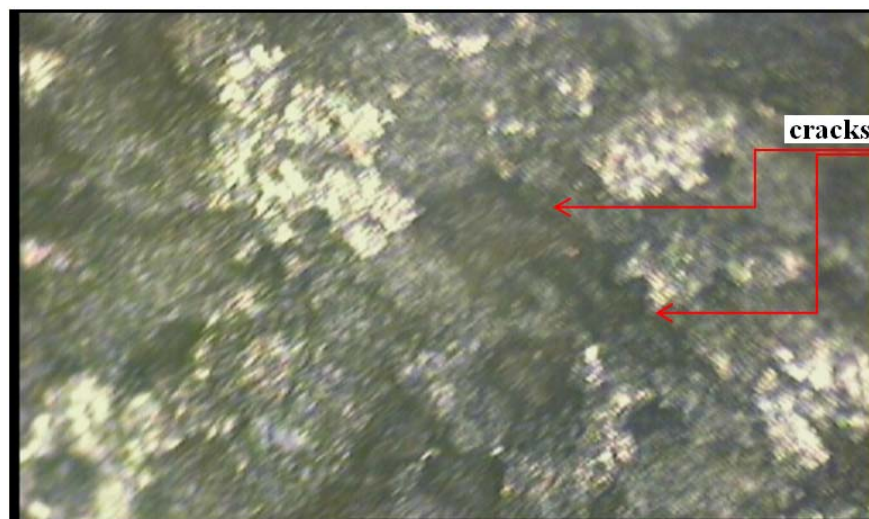
90 min on the structure of the coating layer after heat treatment from repeated tests. The 90 minute heat treatment is the best way to improve the cermet coating layer.



**Fig. 4:** Microscopic structure of coating layers after thermal treatment prepared at different distances (a) 8 cm, (b) 12 cm, (c) 16 cm and (d) 20 cm.

From this figure, we notice that different and sintering process of the coating layer and improvement the physical properties with increasing temperature and we found that the optimum spraying distance is 12cm derailing upon the homogeneity of layer coating and hardness value. Fig.5 illustrates the thermal treatment sample at 1150 °C for 90 min, we observe

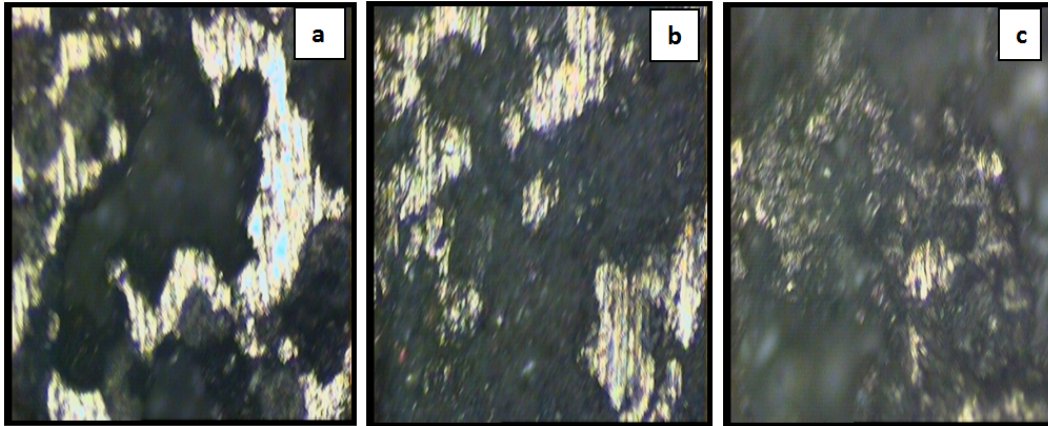
surface defects, dislocations, and porosity which thermal treatment never forbade it well as well as cracks happened. These cracks are called the vertical cracks which begin from coat layer and extended vertically inside and oxygen is coating so the thermal treatment at 1000 °C provides good physical and mechanical properties.



**Fig.5:** Thermal treated at 1150 °C, microscopic structure of cermet coat after laser treatment.

After placing adjust the spray gun at distance 12 cm as the best distance with a coating layer with thickness  $(1.35 \pm 0.3)$  mm of a good adhesion force. The prepared samples at a 50cm distance for 10 sec treated by laser at (400, 460, 500) mJ energy. The

microscopic pictures show that the depth of molten layer proportion to laser beam energy associated with diffusion and grain softening process, in addition to occurs oxidation regions as shown in Fig.6.



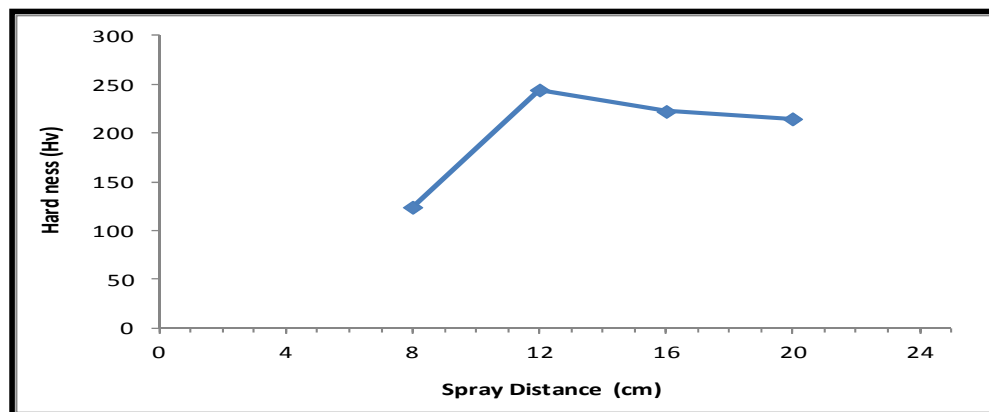
**Fig.6: Coating layer after Laser treatment at different pulse energies (a) 400 mJ, (b) 460 mJ and (c) 500 mJ.**

The coating layer after treated thermally, are – the molten process has been occurring and especially the regions of big grain size, which in turn, leads to softening the grains and diffused there as we are many surface defects has been removed after treated with laser-like unmolded grains. The surface harshness was lesser than the other energies which present result in good agreement with that of [10]. which studies the thermal treatment by

laser and illustrating the laser effect on the surface coating layer (Ni-Cr-Al-Y).

#### **The effect of the thermal treatment on the hardness of cermet coating**

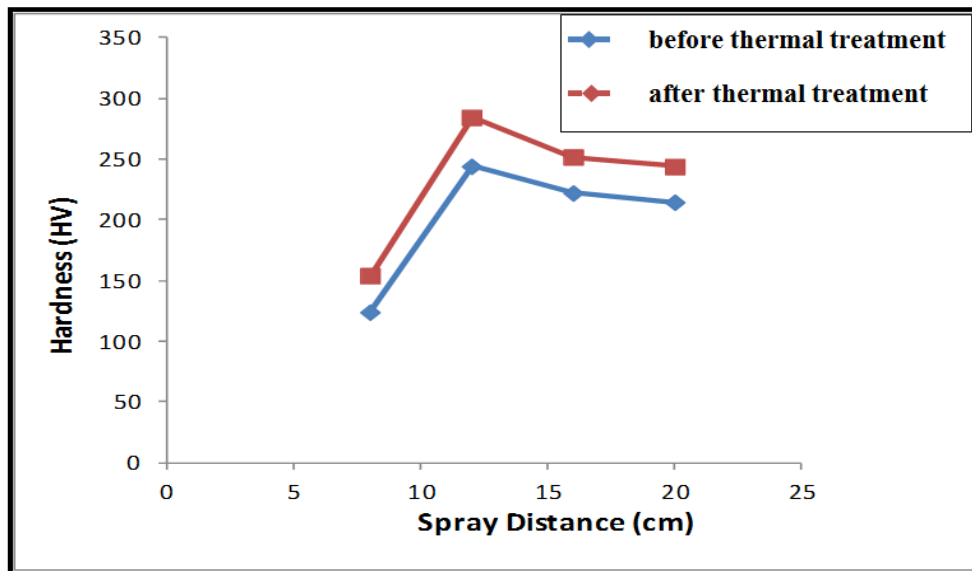
Through Review the obtained result of samples hardness at spraying distance, we note that the best value of hardness occurs at distance 12 cm, where the hardness value equals to 244.2  $H_v$  and Fig.7 illustrates the hardness relations at various spraying distance before thermal treatment.



**Fig.7: Hardness relation with spraying distance before thermal treatment.**

From the above figure, when the spraying distance is close, the hardness will be small, then the hardness will increase to the optimum value the distance by increasing the spraying distance. The above figure is in accordance with the microscopic structure in the Fig.4(b) where the best microscopic structure a separation spray distance equal to 12cm where the coats are homogeneous and more compact in comparison to the other microscopic structure at different spray distance, which contains many surface defects and this equal with the result of

the optical microscopic Test the when we treating the samples thermally for samples at the same distance for 90 min and temperature 1000 °C, we observe huge change in Hardness value for the layer hardness value of the cermet coating, where the value increases to 284.5 Hv as shown in Fig.8, and this belongs to increasing the binding among the atoms of the coating layer due to heat, which will leads reducing porosity and then the hardness of the cermet coating layer will increases.



**Fig.8:** The variation of hardness with spraying distance before and after thermal treatment.

The hardness value is increased after laser treatment incredibly, where we take the hardness value at some spraying distance (12 cm) after treating with laser at different energies (400, 460, 500) mJ are shown in Fig.9. The increment in hardness value is actually

and this produced from removing all crystal defects, holes and porosity, where a re-molten process occurs and that the hardness value increases due to the improvements of structural of coating layer [15].



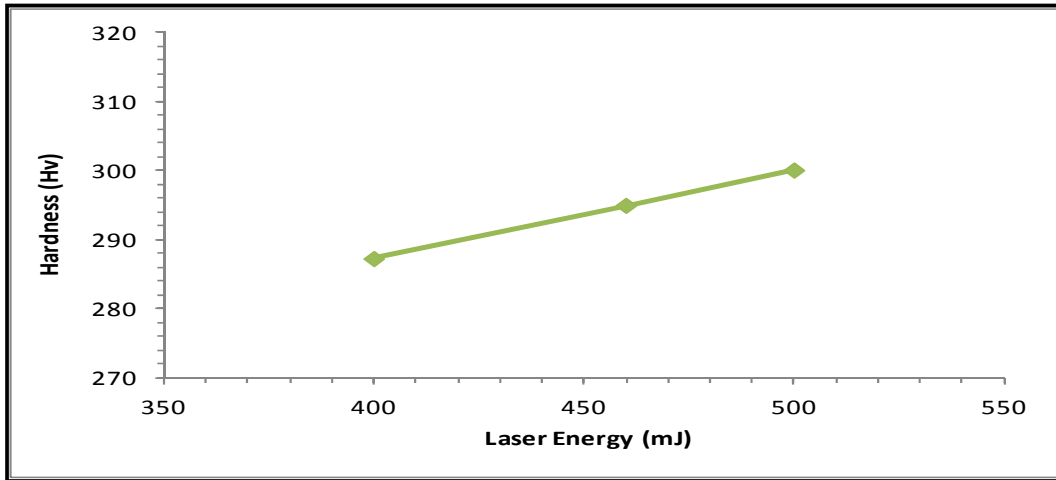


Fig.9: The hardness value with laser energy at spraying distance (12 cm).

**Thermal treatment effect on the adhesion forces of cermet coating**

We can define the adhesion force is that an equivalent force which required to dislocate unit area from the coating layer from the base or are a binding force between the base surface and the cermet layer base. We used six measurements at spraying distance

(12 cm) only, and these measurements taken at different temperatures (700, 800, 950, 1000 and 1100) °C. These result show 5.16 MPa adhesion force before thermal treatment after that, we obtain an increase with thermal treatment to reach its maximum value at 1000 °C as shown in Table 2.

Table 2: Variation of coting adhesion forces with thermal treatment.

No. of Sample	Thermal treatment °C	Adhesion coated layer (MPa)	Type failure
1	700	5.6	adhesion
2	800	7.1	adhesion
3	950	8.7	adhesion
4	1000	10	adhesion
5	1100	6.5	adhesion

Fig.10 represents the relationship between adhesion of the coating layer with treating temperature. The benefit of the thermal treatment is that lead to lower the porosity and nodes of the coating layer and the increased adhesion force due to improvements the structure construction of coating layer, but the samples which treated thermally at 1100 °C, it adhesion force are reduced by a great amount are shown in Fig.10 below and this be due

to the oxidation process occurred for the coating layer when exposures the high temperature in un-vacuumed region and suffering from cracks and dislocations in the coating layer are it clear from the microscopic structure test. The samples are suffering from adhesion fail in the boundary line between the coating and the substrate and this in good agreement with that of [16].

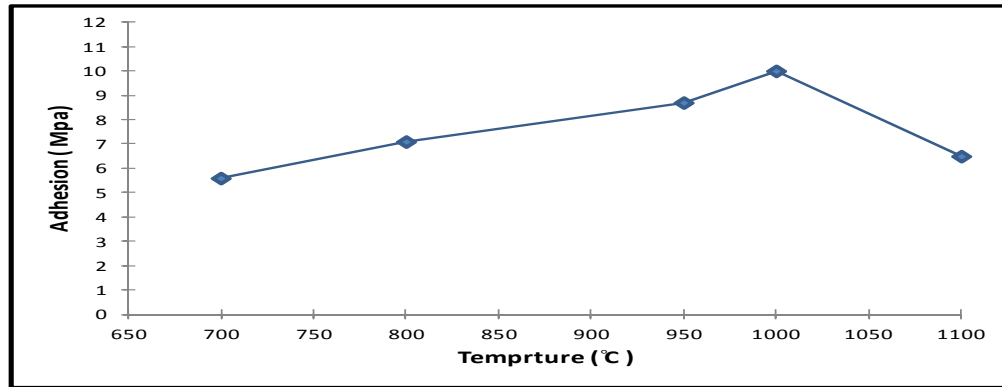


Fig.10: Relationship between temperature and adhesion of coating layer.

### Conclusions

- 1-The optimum distance between the nozzle of spraying gun and the base surface is 12cm for obtaining the best layer.
- 2-A coating of high stiffness, adhesion is excellent homogeneity.
- 3-The coating layer features are improved and get better after accomplishing the thermal treatment Process at 1000 °C in 90 min. in this case the Hardness of coating layer will increase from the value to 244.2 HV T to 284.5 Hv T.
- 4-The Hardness value is increased after exciting the layer treatment. At the ideal spraying distance to the value 300 Hv T.
- 5-The adhesion force is improved between the base surface and the laser of the composite mate with Temperature increase the adhesion force from the value 5.16 MPa to 10 MPa at 1000 °C.
- 6-The laser treatment for the spraying layer by flame to the improvement of the composite mate.
- 7-The most laser treatment obtained at the energy 500 mJ, which provides it the max. Hardness and best properties.

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