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# Factors affecting irradiation of nano & micro materials by laser treatment industrial unit

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**Abstract.** This work presents an industrial unit for hardening and treatment of nano-materials and micrometers to improve their physical, chemical and mechanical properties by exposing them to laser radiation. The treatment process is carried out through the laser treatment industrial unit. It is composed of circular metal base with motor, rectangular holder, moving sides. The sample installation cavities which are metal joints that widen and shrink according to sample dimensions by incorporating the cavities vertically and moving the sides, the unit has black front and back barrier to prevent lasers with bottom cavity to store the black barrier. Scaling must be compatible with laser diameter or width; the unit has partition barriers and fixing arms to control the process. Laser and unit are operated to complete the treatment, method is physical process, the laser beam go through treated material without being absorbed or interact by transparent material, the treated nanomaterials or short fibres were collected. The samples were exposed to Nd -YAG third harmonic generation and Argon ion CW laser. The hardness was measured before and after laser irradiation. The current work will present an application on the hardening of E-fibre glass 731ED 1/32".The change in hardness were explained by raman spectroscopy. The research end with conclusions and recommendation for the expected application of the new treatment unit and method.

**Keywords:** laser, treatment unit, nano-materials, micro material.

## 1. Introduction

The laser is a very powerful and very useful instrument in modern nanoscience and nanotechnology. Laser radiation-based methods play an important role in the development and modification of nanostructures [1],[2]. Nowadays Nanophotonics and Nano optics are the most important study of the behavior of light on the nanometer scale, and the interaction of nanometer-scale objects with light [3],[4].

This science is branch of optics, optical engineering, electrical engineering, and nanotechnology [5]. Laser beam interaction with a particle is the interaction of the laser's electromagnetic wave with the particle's electrons (i.e. absorption and scattering of the laser's photons by the particle's electrons) [6],[7]. the interaction mechanism of the laser beam with nanoparticles is needed to control the laser processing of different nano-objects because only very thin layers of nanoparticles can be efficiently and homogeneously irradiated [8], [9].

Irradiation of micro materials, powders and nanomaterials leads to some phenomena such as recrystallization, segregation, homogenization and relaxation, phase transitions, phase decay and arising, amorphization, sintering and filling of micro- and nanopores (nanocapillars) [10],[11]. All



these processes lead to a nanostructure evolution, so the laser is a very powerful and very useful instrument in modern nanoscience and nanotechnology because it is accompanied with the changes of its physical, chemical, mechanical and other properties[12],[13].The knowledge of the interaction mechanism of the laser beam with nanoparticles is needed to control the laser processing of different nano-objects[14].

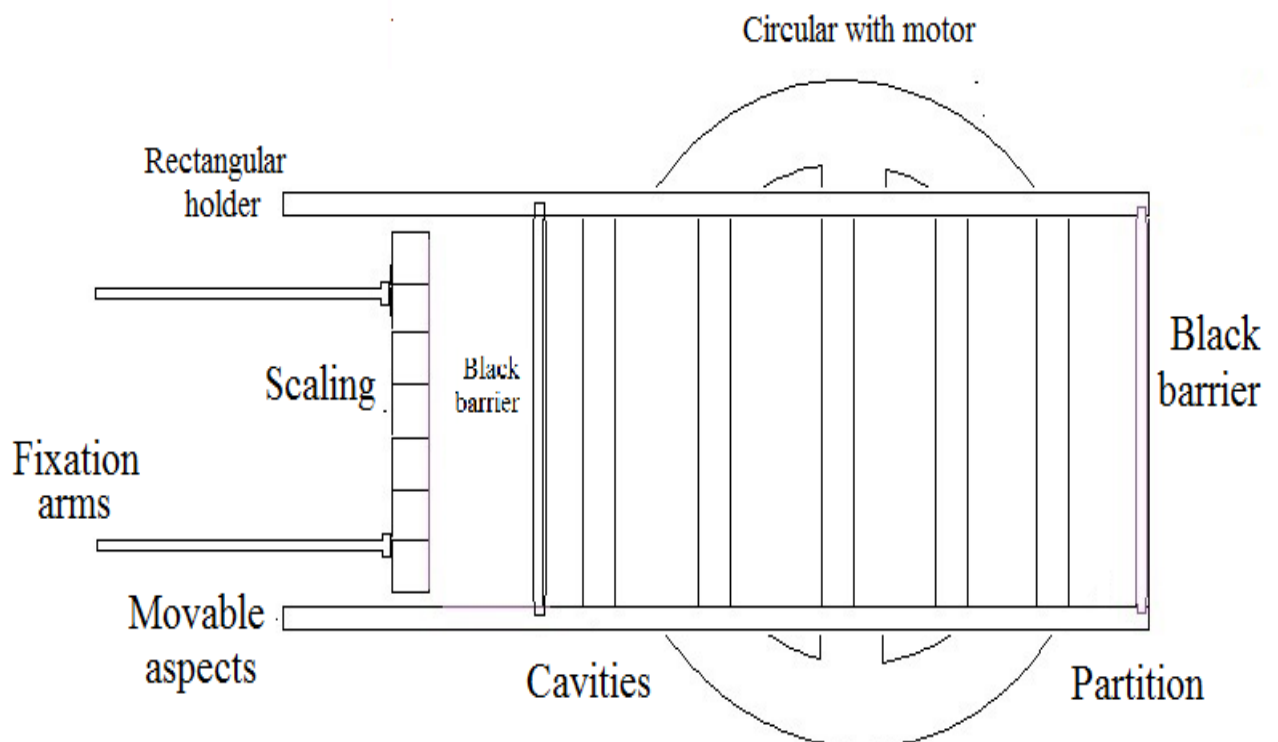
## 2. Experimental work

### 2.1. The machine

The machine used in this work is designed specially for treatment of nano and micro particles by laser on the industrial scale and registered in the Egyptian patent office number 450/2018 [15].

The unit consists of the following parts:

1. Circular hoop with motor which move 360°and
2. Rectangle holder which is divided into cavities to fix samples in front of the laser beam
3. Movable sides to adjust the size of the installation cavities
4. back and forth black barrier to prevent laser beam accidents
5. Bottom cavity to store the black barrier at treatment and allow laser beam transition
6. Gradient scaling to control the movement of samples in front of laser beam
7. Partition barriers can be moved when the sample thickness is increased
8. Rear armrests to prevent movement during the handling of sample which cause the deviation of the laser beam. Figure 1 shows the main construction of the laser treatment unit, while figure 2 shows the simple prototype [15].



**Figure 1.** The main construction of the laser treatment unit [15].



**Figure 2.** The simple prototype of the laser treatment unit

#### Processing and Operation of the machine

1. Circular hoop moves in two directions  $0^{\circ}$  C- $180^{\circ}$ C
2. The mechanical movement of the hoop does not allow tilt angle or inclination may affect the path of the laser beam.
3. The hoop moves by motor and have side arms for manual movement
4. The position of the hoop is adjusted in the front of the laser beam by self-levelling
5. The samples were placed in the cavities in the rectangular holder. The sides were moved to adjust them according to the size of the treated sample
6. arms of the installation moved to close the movement of the sides to confirm the sample in the operating mode.
7. The safety black barrier is adjusted in the black to prevent laser beam crisis [15]

#### 2.2. The material

The work will present an application on the hardening of E-fiber glass 731ED 1/32" with length to diameter ratio  $L/d=50$  (cylindrical particles) and  $L/d=1$  (spherical particles). Table 1 shows the physical properties of the Fiber glass type-E used in this work.

**Table 1.** The physical properties of the Fiber glass type-E.

Property	Dimension	E-type fiber glass
Density	gm/cm <sup>3</sup>	2.58
Refractive index		1.558
Softening point	$^{\circ}$ C	846
Annealing point	$^{\circ}$ C	657
Strain point	$^{\circ}$ C	615

The transparent cover from PMMA was used to hold the particles in the front of the laser beam and dissolved after the process by mixture of organic solvents.

#### Laser irradiation

Samples used in this investigation were in a shape of disk of 25mm diameter and 7mm length. The irradiation is done on both sides of the samples and in different positions to cover all the area of the sample and achieve homogeneous surface suitable for testing. The different lasers irradiated samples at different conditions were examined before and after laser irradiation and the effect of laser was determined.

The type of laser used is:

1. Nd-YAG I used in the third harmonic (wavelength =355nm) which lies in the ultra violet (UV) region with power of bout 10milli watt, duration time 7nsec, repetition rate 17HZ and energy (40mmj/pulse) at different number of pulses.[6]

2. CW argon ion laser (innova400) which have power of 25 watts multi-line wavelength from 514.5 nm to 465.5 nm , the power used in the experiment is 1 watt.

### 2.3. Hardness

The hardness of the specimens were measured by using means of Barcol impression according to ASTM-D2583, the indenter cone with  $26^\circ$  and 0.157mm, the sample must have smooth and polished surface with thickness not more than 1.5mm, the load ranges from 6.8kg to 4.5kg each hardness value is an average of 29 reading for every specimen (5 specimen at each condition were examined). The measurements are good up to  $\pm 6\%$ .

### 2.4. Raman Spectroscopy

The behavior of the material was studied by variation of the modes obtained from FT (IR) Raman spectrum. The results were obtained using Bruker FTIR Raman spectrometer using Nd-YAG laser power of 500MW as source of excitation. After irradiation of sample with lasers the spectra were obtained in the range 400 to 4000  $\text{cm}^{-1}$ . The samples were measured by fixing it to holder and placed in front of the beam [27].

## 3. Results and discussion

The unit used in this work show an easily scalable method for rapid irradiation of powder micro and nanomaterials by laser light formed homogeneously in situ, the new unit is suitable for different industrial application, and transfer laser irradiation process to economic and abundant industrial unit [16],[17].

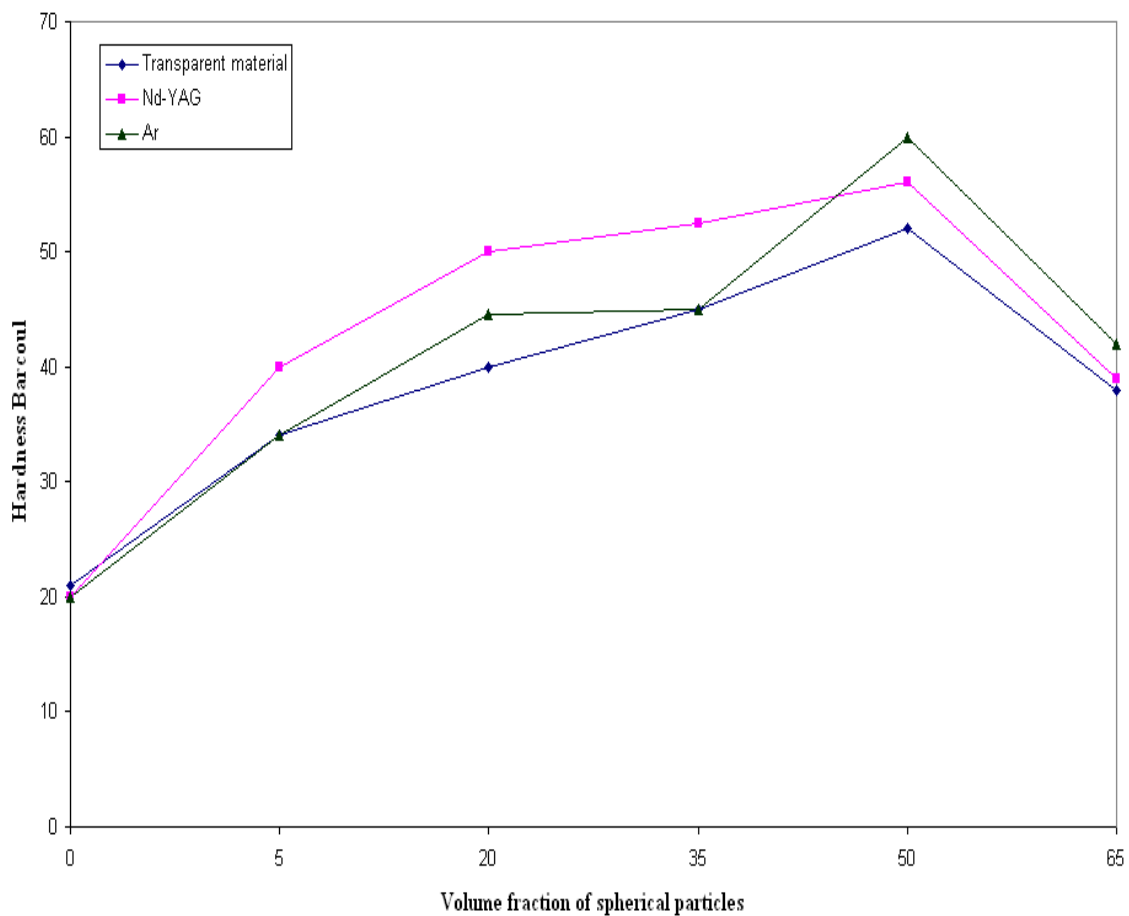
The different parameters which affect the process such as laser type, particle size, volume fraction (number of particles), number of pulses, and surface area exposed to laser irradiation, it has been studied separately to identify the optimum conditions of laser irradiation of micro and nanomaterials by new technique [18].

Figure 3 shows the variation of Hardness for different amounts of fiber glass type -E irradiated at the same length to diameter ratio  $L/D=1$  (spherical particles) by laser pulses at two different conditions of laser irradiation. The first is Ultraviolet laser 355nm with power of about 10milli watt, duration time 7nsec, repetition rate 17HZ and energy (40mmj/pulse). The second is CW argon ion laser (innova400) which have power of 25 watts multi-line wavelength from 514.5 nm to 465.5 nm, the power used in the experiment is 1 watt [19],[20]. In general, the hardness increases with amount of material and laser irradiation, when the amount of material increases the surface area exposed to surface irradiation increases and the amount of absorbed energy increased gradually till 50% volume fraction and maximum value of hardness recorded as 60 barcol. When the number of impeded particles increase overlapping prevent laser irradiation to reach to all parts so the amount of absorbed energy decreased and the hardness value dropped down to 35 barcol as shown in figure (3).

From figure 3 also the improve in hardness for the Nd-YAG treated particles over the Argon ion laser were recorded at the lower concentrations. in general UV laser 355nm low wavelength has higher frequency and higher energy according to the equation [21],[22].

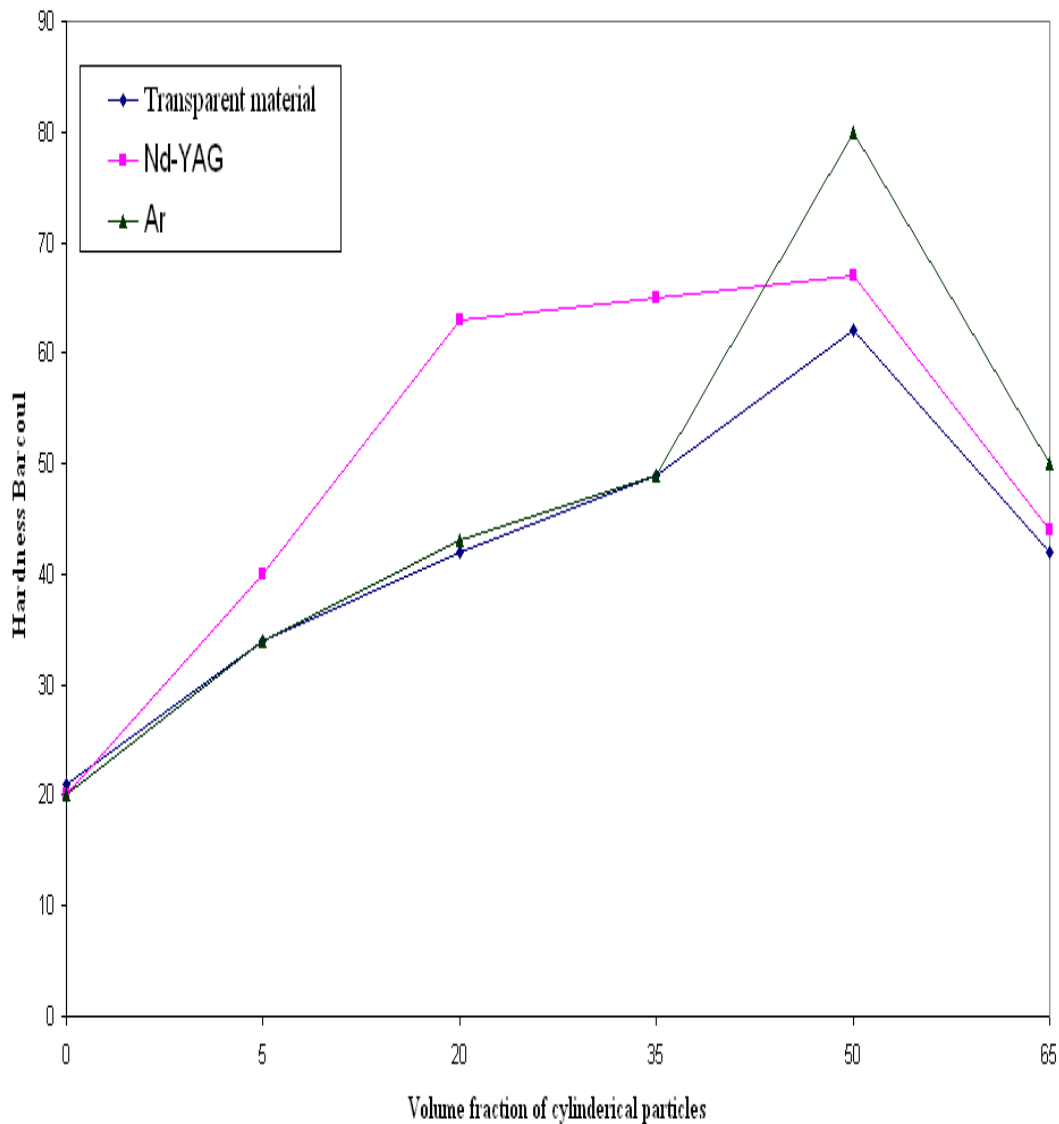
$$E = h\nu = hc/\lambda \quad (1)$$

The samples irradiated by argon laser did not produce any significant effect or pronounced change in hardness value at lower concentrations because the amount of energy absorbed was still low to introduce any structure change, but at higher concentrations %50 volume fraction, the amount of energy absorbed is enough to change the structure of micro and nano particles so the hardness is improved [23],[24].



**Figure 3.** The effect of volume fraction of spherical particles of fibre glass type -E(L/D= 1) on hardness, at different kinds of lasers Nd-YAG 355nm and Argon 507nm.

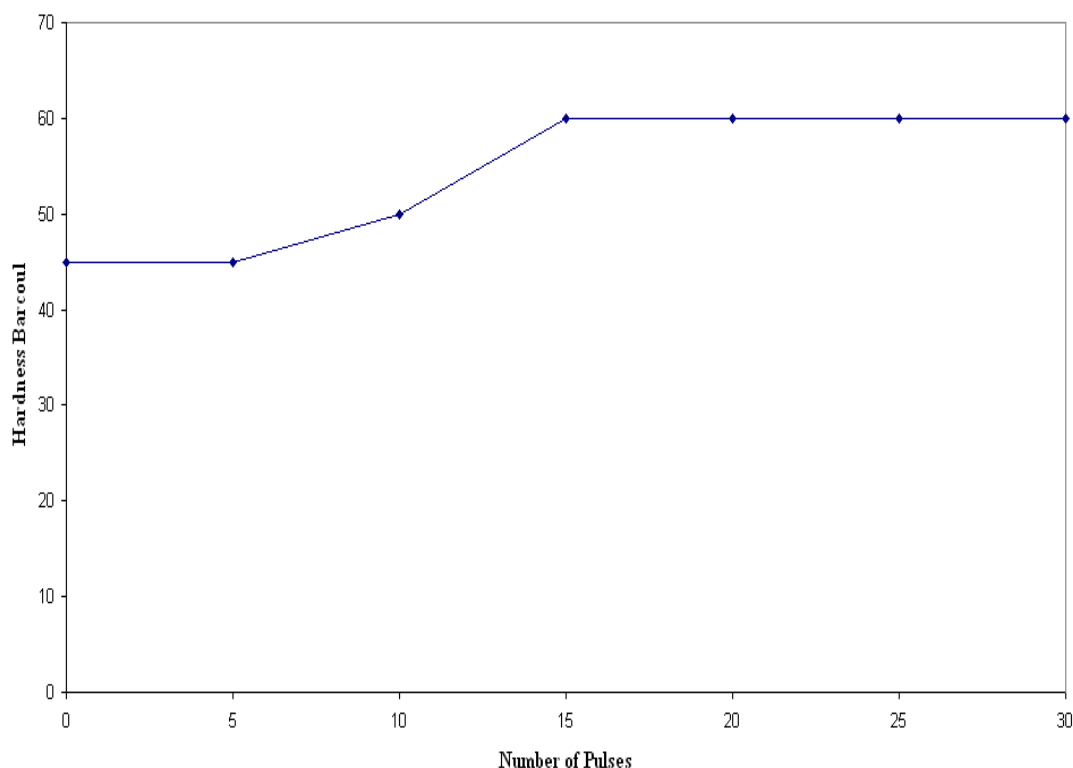
In figure (4) the variation of Hardness for different amounts of micro and nano materials irradiated at the same length to diameter ratio  $L/D=50$  (cylindrical particles) by laser pulses at two different conditions of laser irradiation, all the phenomena recorded in figure (3) was repeated in figure (4) but higher values of hardness was recorded at all conditions for example the maximum hardness value of the irradiated samples was 80 barcol not 60 barcol as recorded in figure (3). The same behavior of the material was happen and the increase of hardness was due to the increase in the amount of laser energy absorbed, the increase in energy absorbed leads to more change in structure and significant increase in hardness, the phenomena of increase in the amount of energy absorbed was happened due to increase of surface area exposed to laser irradiation the cylindrical shape have larger surface area than spherical shape for the same volume[25],[26].



**Figure 4.** The effect of volume fraction of cylindrical particles fiber glass type -E ( $L/D=50$ ) on hardness, at different kinds of lasers Nd-YAG 355nm and Argon 507nm.

To study the phenomenon of laser irradiation the sample has optimum conditions was selected to be tested at different number of laser pulses to show the effect of increase and decrease in laser energy on the change of mechanical properties due to structure change. The hardness values of the sample has length to diameter ratio  $L/D=50$  (cylindrical sample) and volume fraction amount 50% was recorded at different number of laser pulses 0,5,10,15,20,25,30, as shown in figure (5), the improvement in hardness value begins at 10 pulses and decreases gradually with the increase in number of pulses, after that the improvement in hardness stop with increase in number of pulses, at lower number of pulses the energy absorbed does not reach the minimum limit to cause structure changes leads to improve in mechanical properties, after that the gradual increase in number of pulses which mean gradual increase in the amount of energy absorbed leads to gradual change in structure and mechanical properties.

When the samples reach the saturation limit, the change in structure and mechanical properties remain unchanged [27],[28].



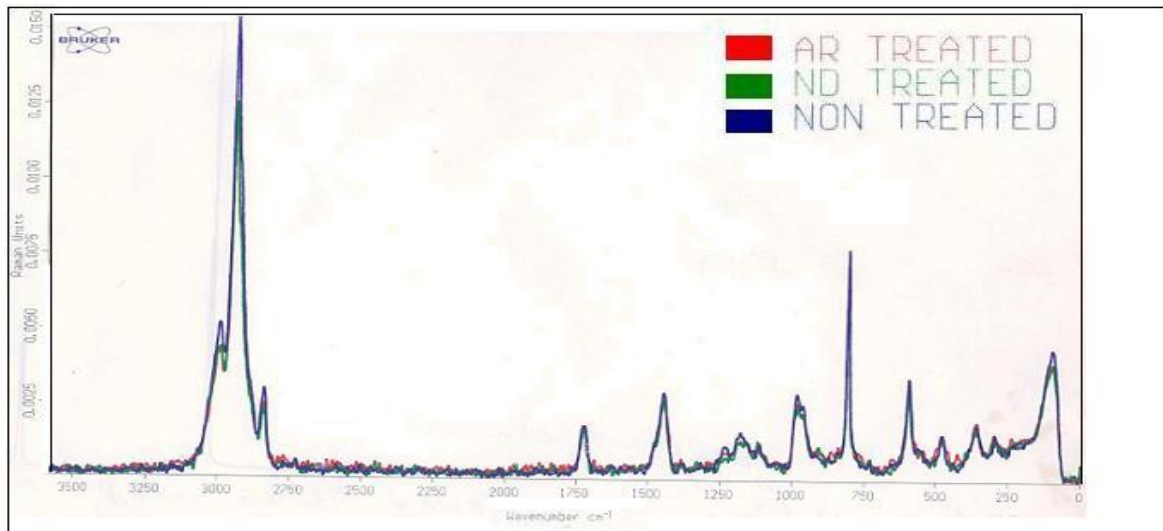
**Figure 5.** The relation between hardness Barcol and number of laser pulses for Nd-YAG 355nm at 50% volume fraction, L/D=50.

The raman spectroscopy can be applied for understanding the mechanism of such complicated processes, the band shape, intensity and center of mass can give indication about the change in the structure due to absorption of laser energy. The most important phenomena observed on the raman spectroscopy were integral intensity rather than peak depth and the width of the band at its half depth. Figure 6 shows the raman spectrum for transparent material before and after laser irradiation by Nd-YAG 355nm and Argon laser 507nm, in this curves there was no change can be recorded due to laser irradiation the transparent media did not absorb any types of the laser used in such study at the selected wavelength, the transparent material can be considered as ideal media to study the mechanism of laser interaction with micro and nano materials at the selected wavelengths[29],[30].

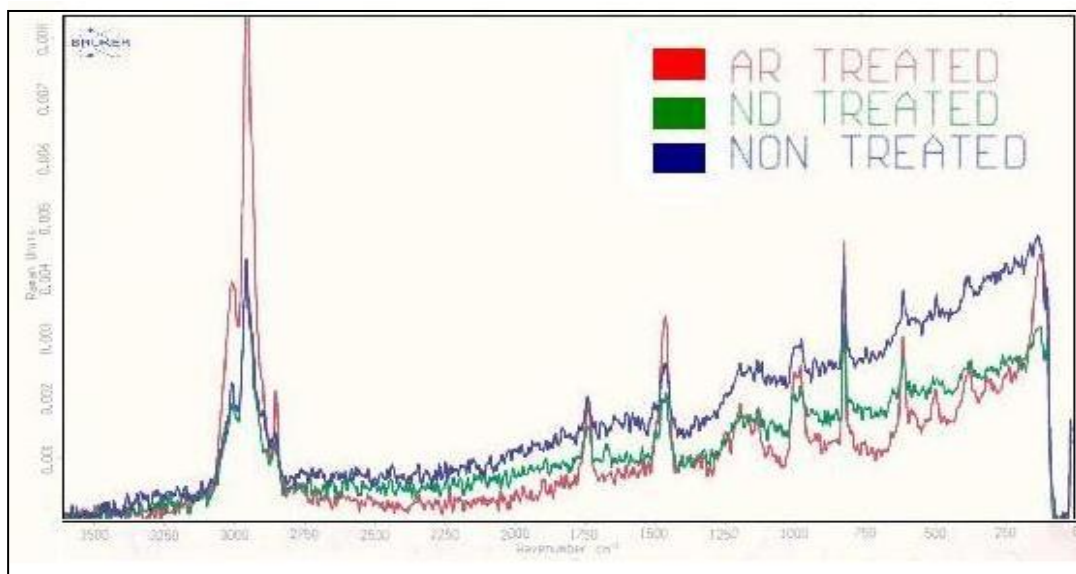
Figures 7 and 8 show the raman spectrum for spherical (L/D=1) and cylindrical (L/D=50) micro and nano particles before and after laser absorption Nd-YAG 355nm and Argon laser 507nm, at 50% volume fraction. A Raman spectrum features a number of peaks, showing the intensity and wavelength position of the Raman scattered light. Each peak corresponds to a specific molecular bond vibration, It is based upon the interaction of light with the chemical bonds within a material, from the above figure the change in peak width and intensity, which can then be used as a quantitative measure of crystallinity, phase and structure changes. There is no shift in peak position which give indication that the chemical composition did not suffer from any significant change [31]. The maximum value of peak intensity was recorded for the transparent materials was 0.0150 raman unit for all irradiated and non-irradiated samples. The maximum value of the irradiated samples was recorded for Argon-ion



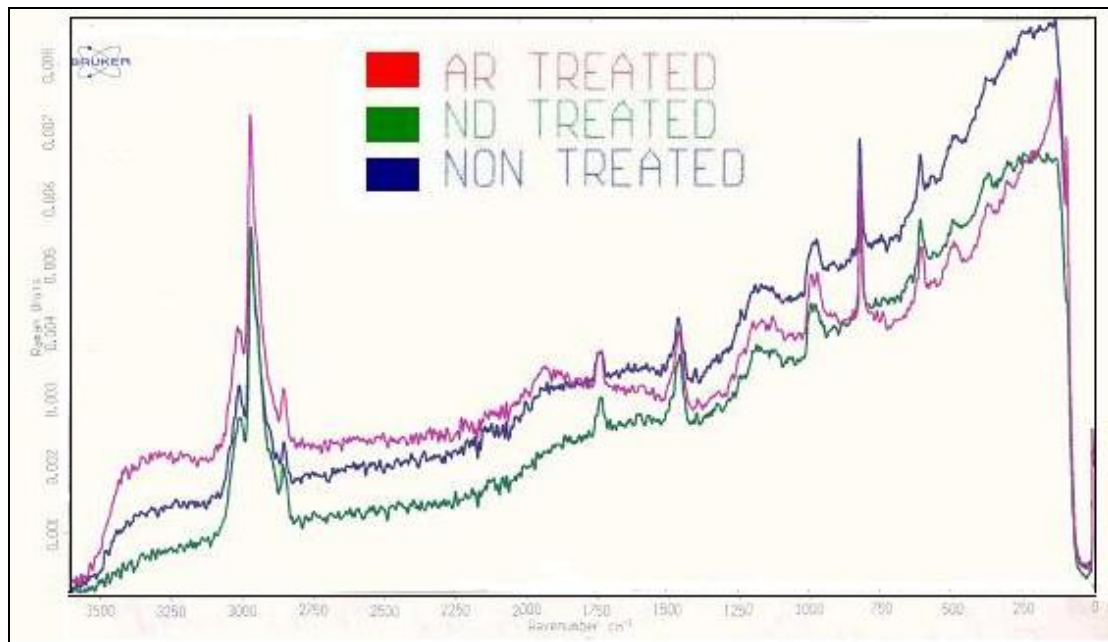
lasers in the range from 0.007 to 0.008 raman unit. The laser irradiation of nano and microparticle by using indirect exposure unit is physical process and suitable for different applications.



**Figure 6.** The raman spectrum for transparent material before and after laser irradiation by Nd-YAG 355nm and Argon laser 507nm.



**Figure7.** The raman spectrum for spherical particles fibre glass type -E, (L/d=1) before and after laser absorption Nd-YAG 355nm and Argon laser 507nm, at 50% volume fraction.



**Figure 8.** The raman spectrum for cylindrical particles fiber glass type -E, ( $L/d=50$ ) before and after laser absorption Nd-YAG 355nm and Argon laser 507nm at 50% volume fraction.

#### 4. Conclusions

1. The new industrial unit is developed to treat the micro and nano material by laser
2. The main factors affecting the laser irradiation of nano and micro materials are:
  - The Laser type and wavelength
  - The number of laser pulses
  - Shape and size of the treated micro and nano particles
  - Volume fraction of micro and nano materials
  - Surface area of the micro and nano particles exposed to laser irradiation.
3. The amount (**volume fraction of the materials which leads to increase in surface area exposed to laser irradiation**) of micro and nanoparticles impeded in the transparent material must be in the range from 35% to 50% volume fraction to achieve optimum laser exposure to the laser beam, and avoid overlapping of the particles which reduce the amount of laser energy absorbed.
4. In general UV laser Nd-YAG 355nm at low wavelength and higher frequency and higher energy rather than visible light Argon ion 507nm, so the samples irradiated by UV laser record improvement in hardness at lower energy levels.
5. The industrial unit for laser irradiation is suitable for different applications
6. The raman spectroscopy explain the phenomena of laser interaction with micro and nanoparticles. A Raman spectrum features a number of peaks, showing the intensity and wavelength position of the Raman scattered light. Each peak corresponds to a specific molecular bond vibration, It is based upon the interaction of light with the chemical bonds within a material,
7. The change in peak width and intensity, which can then be used as a quantitative measure of crystallinity, phase and structure changes. the band shape, intensity and centre of mass can give indication about the change in the structure due to absorption of laser energy. The most important phenomena observed on the raman spectroscopy were integral intensity rather than peak depth and the width of the band at its half depth.

8. There is no shift in peak position which give indication that the chemical composition did not suffer from any significant changes.
9. The laser irradiation of nano and microparticle by using indirect exposure unit is physical process.

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