International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN: 0974-4290
Vol.7, No.5, pp 2181-2193, 2014-2015

Effects of Nd: Yag Laser Shock Processing on The Microstructure, Fracture Surface Characterization and Mechanical Properties of Nickel Chromium and Cobalt Chromium Alloys

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Abstract: The influence of the Nd:YAG Laser Shock Peening (LSP) on the microstructure, micro hardness and mechanical behaviour of Ni-Cr and Co-Cr alloys used for dental prostheses have been studied.. The major clinical disadvantages of the Ni – Cr and Co –Cr alloys are their hardness, lack of adequate ductility and yield strength. These properties combined made finishing, polishing, burnishing of conventional base metal alloys rather difficult. Laser shock processing is an innovative surface treatment, for Ni –Cr and Co – Cr alloys for improving workable mechanical properties and used as substitutes for dental gold alloys. The resultant change in microstructure, chemical composition and grain structure were studied by scanning electron microscopy, Energy Dispersive Spectroscopy and X-ray diffractometry. The mean values of % elongation of Ni-Cr alloys were higher (doubled) due to grain refinement imparted, and the mean values of ultimate tensile strength, yield strength, modulus of elasticity of alloys were significantly lowered after laser shock preening (LSP). On the other hand the experimental results of Co – Cr alloy showed changes in mechanical properties, modification in microstructure and surface hardness. After shock peening, hardness of Ni-Cr and Co-Cr alloys have increased marginally by 17.29% and 3.69% respectively. The mean values of percentage elongations of Ni-Cr increased by 200% after laser peening. It is evident from the above experimentation (LSP), increase of ductility of Ni – Cr facilitates workability, which could produce more reliable removable partial denture (RPD) metal frameworks compared to Co –Cr alloy.
Keywords: Nd:YAG Laser; Laser Shock Peening(LSP); Nickel- Chromium (Ni-Cr); Cobalt-Chromium (Co-Cr); Student’s-t-test; Dental Prostheses.

1. Introduction

Need for the alternate material to gold arise due to the fluctuating market conditions and cost in dental prosthesis. The use of the gold in dental prosthesis gradually reduces over the years due to continuous cost increasing trend. The search and use of the equivalent alternate material enhances for partial removable dentures. Number of alternate materials like commercial pure titanium, titanium alloys, nickel –chromium, and cobalt-chromium alloys are developed and practiced. The major advantage in gold is the recast ability and biocompatibility. Titanium finds application in the place of gold due to low density and comparable strength. But the cost of the titanium and complex workability in the laboratory leads to go for other materials[1-3]. Hence over the year’s use of base metal alloys have come into existence for economic reasons without diluting the dental application requirements of end user. Ni-Cr and Co- Cr alloys became substitutes for noble alloys for dental cast restorations. The versatile properties include mechanical properties, physical properties, corrosion and easy flexibility in manufacturing practices. The properties like abnormal hardness, lack of ductility in “as cast” Ni-Cr and Co-Cr alloys are difficult to deal with in the laboratory [4 – 6]. It is observed that the matrix microstructure of these alloys in “as cast” condition found to be responsible for the inherent properties. The “as cast" microstructure is dominantly dendritic with different sizes of primary and secondary arms and in-homogeneity metal matrix. The microstructure need to be modified or re-crystallized to overcome the complex properties and to achieve the desirable metal framework. Laser shock peening is one of the cost effective surface treatments method which can change the morphology of the metal matrix and mechanical properties. This treatment causes strain hardening, compressive residual stresses and microstructure changes from "as cast" to desirable and workable metal frameworks.

Laser peening is an innovative commercially–available surface enhancement process for increasing resistance of the components in major properties. This process creates residual compressive stresses deep into part surfaces, typically 5-10 times deeper than conventional metal shot peening. The compressive stresses inhibit the initiation and propagation of fatigue cracks. The shock peening drives a high amplitude shock wave into a material surface using a high energy pulsed laser. When high power Q-switched and low losses, Nd: YAG laser surface treatment is applied to cast Ni-Cr and Co – Cr, metal frameworks, it is expected that these metal frameworks will have sufficient hardness and improved yield strength and adequate ductility to withstand mastication stresses [7-13]. Therefore, the purpose of this study is to investigate the mechanical properties, by analyzing their surface properties such as Vickers micro hardness, microstructural characterization of cast-specimens of nickel – chromium (Ni-Cr) and Co-Chromium (Co-Cr) laser treated with Nd:YAG laser and compare the results with untreated specimens.

2. Materials and methods

2.1 Specimen preparation

Cylindrical (10mm x 10mm) specimens and dumbbell - shaped tensile specimens of (ISO 6871-1: 1994, gauge length 18 mm and diameter 3 mm were prepared as shown in figure 1 for casting of a commercially Nickel –Chromium (Ni-Cr) alloy and Cobalt - Chromium (Co-Cr) alloy and their material specifications are shown in table 1 and 2.

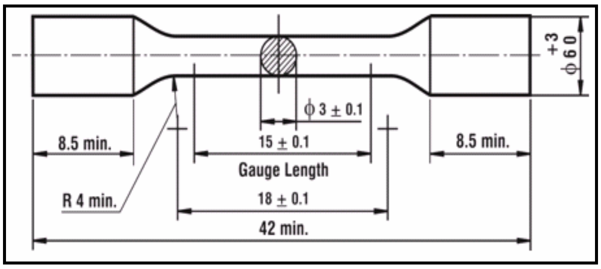


Figure 1 Test specimen was fabricated according to specification of ISO 6871 for dental base metal casting alloys (all dimensions are in mm)

Table 1 Material specification: Ni-Cr alloy (% in weight).

Elements				
Supplier: Bego Bellabond	Ni	Mo	Cr	Others (Beryllium free)
ISO 9693/ISO 22674	65.2	9.5	22.5	Fe, Si, Mn, Nb 2.8%

Table 2 Material specification: Co –Cr alloy (% in weight).

Elements					
Supplier: Wirobond C Bego Bellabond plus	Co	Cr	Mo	W	Others (Nickel & Beryllium free)
ISO 9693/ISO 22674	61%	26 %,	6%,	5.0%	Fe, Si, Ce 2.0 %

These alloys were cast with Phosphate –bonded investment materials (Bellasun, Begosol (Bego, Germany) using an Induction melting centrifugal casting machine (Technico, Germany). The burn-out schedules for the investment materials and casting procedures follow the manufacturer's instructions. After casting, the moulds were bench cooled to room temperature and tensile specimens were retrieved. The surfaces of the tensile specimens were air-abraded with 50µm, $Al_2 O_3$ abrasive particles to make uniform surface conditions. The specimens were then sandblasted (make: Bego sand blaster) to obtain clean surfaces.

2.2 Laser treatment procedure

The laser treatment parameters were carried out on blank cylindrical specimen identical to tensile specimen. Laser treatment for cast - Ni-Cr and Co –Cr alloys was conducted using Nd: YAG laser Machine (Lee Laser system, 7605 Presidents Dr. Orlando, FL 32809, United States) under the manufacturer's and process conditions as in table 3.

Table 3 Laser process parameters

Spot Diameter	Laser energy density	Pulse frequency	Peening speed	No. of scan per second	Argon shielding Nozzle angle
1.2 mm	450 J/cm^2	1.1KHz	160 mm/s	4	45º

The Laser Peening creates shock waves at the metal surface of specimens (Gauge length area), which intense drive compressive stresses into the metal to depth approximately 0.48 mm to 0.73 mm deep. During laser treatment, argon gas shielding was applied from two nozzles set at a 45º angle on both sides above the treatment area of the specimens and all the process principles of laser transformation hardening are shown in figure 2a to 2g [14-21]. ***M.Rozmus -Gornikowska*** in his study[22], the surface of the treated materials can be coated with sacrificial layer (blackpaint) (to minimize undesirable thermal effects on the surface caused by the laser) as a laser energy absorbing medium and covered with a transparent layer as water. When the laser beam strikes the coated material, the black paint, due to absorption of laser beam energy, is heated and instantaneously vaporized. The vapor absorbs the remaining laser beam radiation and produces plasma. The rapidly expanding plasma creates a high surface pressure, which propagates into material as a shock wave. The shock wave can induces compressive residual stresses that penetrates beneath the surface and strengthens the surface of the treated material. ***A.Kruusing[23]*** carried out a study by using a material that is transparent as water to the laser beam, the expansion of this hot plasma can be delayed compared to the free expansion that occurs in direct ablation and thus the magnitude of the pressure can be controlled. Once the peak pressure exceeds material yield strength, the transient shock pressure causes severe plastic deformation, microstructural changes. high increase in dislocation density, refined grain size, influence of roughness surface, compressive residual stresses, and increased hardness at the surface and in the subsurface. As a result, the mechanical properties on the work piece surface are enhanced to improve the performance of fatigue, wear, corrosion and foreign object damage.

To ensure the entire testable surfaces, 60% overlapping procedure was followed. Laser treatment was also adopted for the holding areas of the specimens. Cast- Ni-Cr and Co –Cr alloys of specimens were also reserved as control specimens.

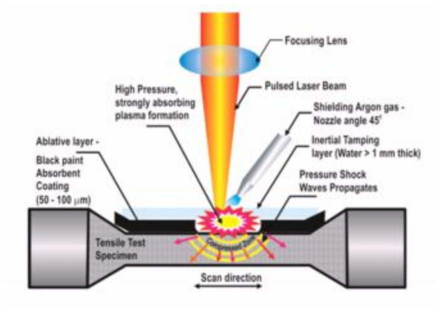


Figure 2a Mechanism for generation of compressive residual stresses improvement using laser irradiation

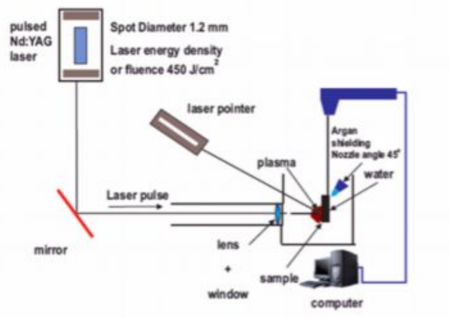


Figure 2b Laser shock peening (LSP) experimental setup

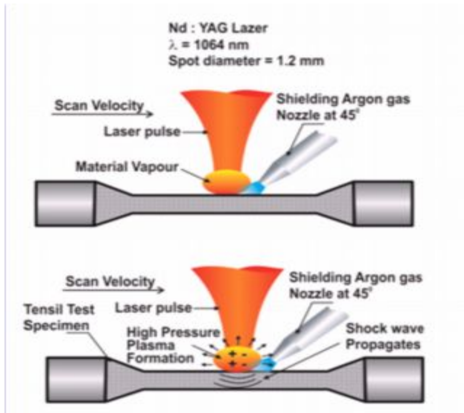


Figure 2c Direct ablation

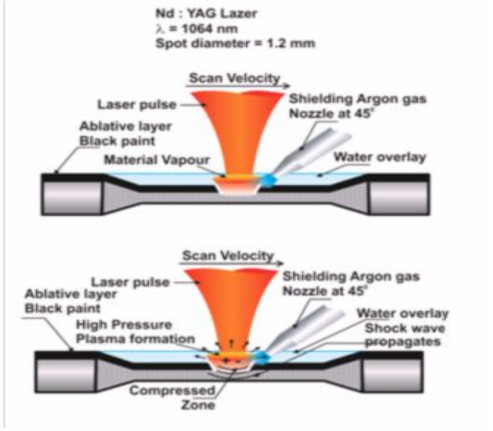


Figure 2d Confined ablation

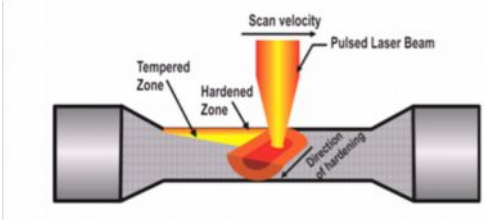


Figure 2e Cross section of tensile test specimen

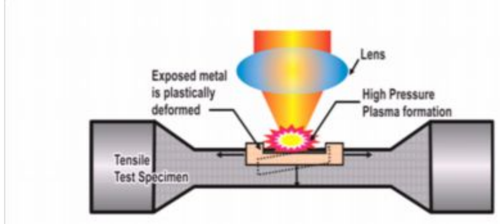


Figure 2f After laser irradiation

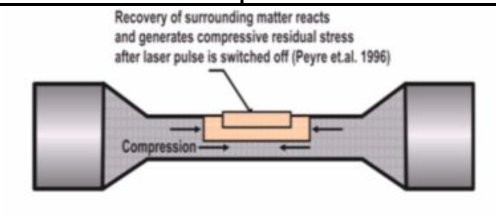


Figure 2g Stretching of laser pulse irradiation

2.3.Microhardness measurement

The Vickers micro hardness depth profile were made on the cross-sections of a cylindrical specimens (10 mmx10 mm) for both the Ni-Cr and Co-Cr alloys were determined (*ASTM E384 – 11e1)* with a load of 500 grams (4.905N) applied for 10s (micro hardness tester (Model: Wilson Wolpert, – Germany). The measurements were conducted from 25 µm from the cast surface to 450 µm in depth. The load of 500g was selected as the hardness was at a higher level. Five measurements were averaged for each depth.

2.4 Tensile testing

Tensile testing was performed using a tensometer of capacity 5 KN with online stress Vs strain graphical mode (software based). In order to evaluate the consistency of the test results, five test specimens in each category were tested for both the untreated and laser treated alloys. Ultimate tensile strength (UTS), yield stress (YS), percentage of elongation and modulus of elasticity were recorded and a strain rate of 1.25mm/min was maintained. The means and standard deviations of each property were calculated.Data (n =5) were statistically analyzed by ANOVA and Tukey's test ($p<0.05$) and the figure 4a and b shows the fractured tensile test specimens of Ni-Cr and Co-Cr alloys respectively after tensile testing. The mean values of Ni-Cr and Co – Cr alloys of specimens of ultimate tensile strength (UTS), yield strength (YS), modulus of elasticity (E) are significantly decreased after laser shock peening as shown in fig.5 (a) &(b) and 6 & 7.

Fig. 4a Fractured tensile test specimens of Ni-Cr alloy after tensile testing

Fig. 4b Fractured tensile test specimens of Co-Cr alloy after tensile testing

2.5. Material characterization

To determine the compounds formed during surface treatments, X-ray diffraction (XRD) was carried out on a 2θ (degree) reflection geometry using CuK_{α} radiation (Model: 3003 TT, GE Inspection technology, Germany). The Cu X-ray tube voltage was set to 40kV, tube current to 30mA, a 2 theta scanning rate of 0.04°/sec and analysis region of size $0.5cm^2$. SEM fractography and Energy Dispersive Spectrometry - EDS (Model: Carl Zeiss MA15/EVO 18, Scanning Electron Microscope, Resolution of 3.0 mm at 30 kV with SE detector) have been carried out for untreated and laser treated specimens of Ni-Cr and Co-Cr alloys after tensile testing to ensure the microstructure of cast surfaces and quantitative element analysis.

3.0 Results and Discussion

3.1 Tensile testing

The experimental results of the tensile test carried out *on* Ni-Cr alloy before and after treatment shows reduction in UTS & YS with substantial increase in % elongation. On the other hand the effect of LSP on Co-Cr resulted in marginal reduction in the UTS &YS and increase in % elongation. This change is due to the LSP effect which had changed the cast microstructure to more uniform homogenized matrix. A good increase in elongation shows the effect of LSP. The dendrite pattern of austenite changed into fine crystalline. The effect of laser peening lowered the UTS, YS and MOE. The re-crystallization of the sub-surface grains resulted in the homogeneity of the matrix by changing the inter–dendrite grains. This is evident from the enhanced % elongation observed. The lowering of YS value with double enhancement of elongation after laser peening could allow greater cold work capability (workability) in dental restorations. However, the above effect is less pronounced in the Co –Cr alloy. As cast Ni –Cr alloy generally would result in lower elongation due to the inter-dendritic pattern of austenite γ - grains. In case of Co – Cr such change in the microstructure is less pronounced. But the elimination of dendrite coring has contributed to marginal improvement. Phenomenal increase in elongation of Ni – Cr after laser peeing would result in adaptability of materials to thinner, lighter and long span bridging dental applications. The lowering of MOE indicates relative lowering of stiffness of the alloy facilitating the easiness of the restorations. The comparison of YS, MOE, and percentage of elongation is more favorable for dental restorations to Ni-Cr materials. The experimental determination of mechanical properties of Ni –Cr and Co – Cr alloys showed that Ni –Cr has positive trend compared to Co – Cr alloy which has very high YS, MOE, and lower elongation as shown figure 7. The ranges of modulus of elasticity of Ni- Cr alloy are from 150 to 250 x 10^3 MPa whereas Co –Cr is 450 x 10^3 denotes the stiffness of the alloy. Base metal alloys are twice as stiff as the gold ceramic alloys. Thus, casting can be made thinner, thereby, decreasing the weight of the RPD (Removable partial dentures). Practically thinner, lighter castings and long span bridges can be made, where other metals are likely to fail because of 'flexing'. Gold alloys require a minimum thickness of at least 0.3 to 0.5mm, whereas base metal alloys like Ni-Cr, Co-Cr alloys, copings can be reduced to 0.3 mm or even 0.1mm claimed **[21].** The yield strength of given alloy determines its resistance to permanent deformation.

It is important that dental restorations do not deform plastically under masticatory stress. Large restorations, such as long span bridges, are more likely to deform if made of alloys with low yield strength. Therefore, the alloys under investigation appeared to be more advantageous after laser- treatment than gold alloy in this respect. This experimental Ni-Cr and Co-Cr alloys seem to fulfill the criteria as well.

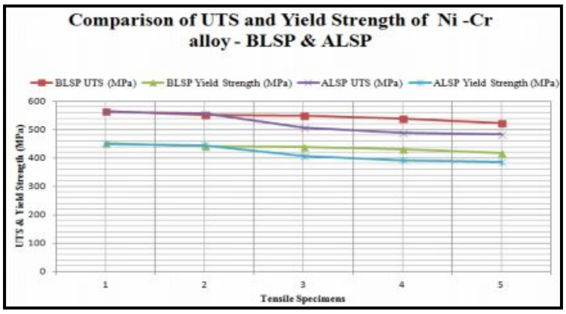


Figure 5a Comparison of Yield strength and UTS for both untreated & laser treated specimens of Ni-Cr alloy.

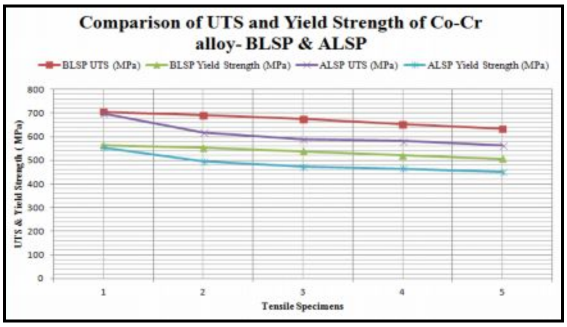


Figure 5 b Comparison of yield strength and UTS for both untreated & laser treated specimens of Co-Cr alloy.

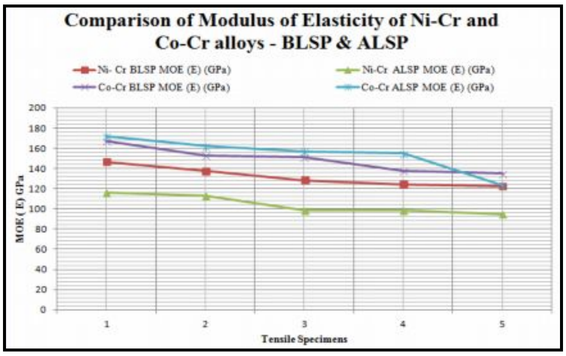


Figure 6 Comparison of modulus of elasticity (E) for both untreated & laser treated specimens of Ni-Cr and Co-Cr alloys.

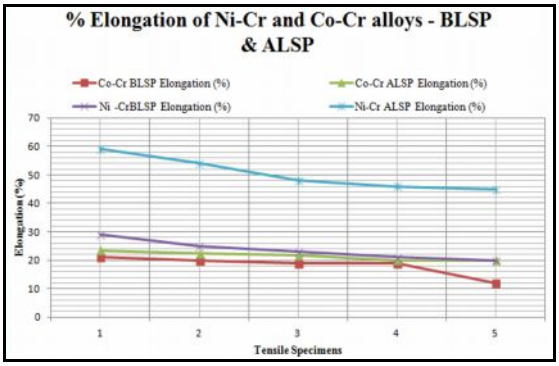


Figure 7. Comparison of % of elongation for untreated and laser treated specimens of Ni-Cr and Co - Cr alloys.

3.2. Hardness depth profiles

Vickers micro hardness depth profiles obtained before and after laser peening from the cylindrical specimens are depicted below. The mean hardness values of BLSP of Co-Cr is higher than the cast surface of Ni-Cr specimens, when compared. Whereas the hardness values of ALSP of Ni-Cr and Co-Cr alloy are marginally higher values at the laser treated surface than the Ni-Cr specimens. But the hardness values of laser treated groups gradually decreased to a value similar to the bulk hardness of the control group at a depths greater than 450µm as shown in figure 8.

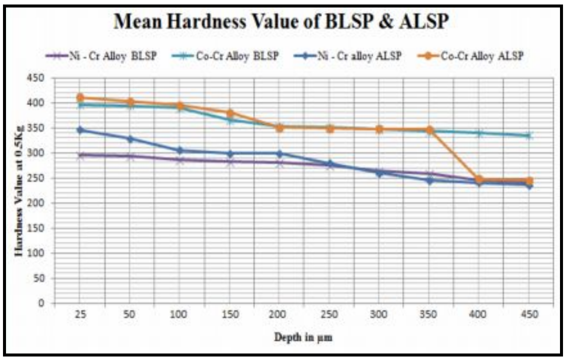


Figure 8 Comparison of hardness depth profiles before (BLSP) and after laser peening (ALSP) at 25µm from the cast surface to 450µm in depth.

3.3 Microstructure analysis of the surfaces

3.3.1 XRD analysis

Figure 9 shows XRD patterns of as-cast Ni-Cr, Co –Cr alloy surfaces, laser treated at 450J/cm^2 with effective argon shielding. The as-cast Ni –Cr was mainly composed of austenite phase (FCC). The crystallinity of pure Co-Cr is higher when compared to that of Ni-Cr alloy. Further, no impurity peaks were observed because of the effective argon shielding environment. But in case of Ni-Cr alloy, complete crystalline peaks observed. The Ni-Cr alloys ($Cr_{1.12}Ni_{2.88}$) crystalline character was matching with JCPDS -65-5559.

3.3.2 Microstructure analysis

The "as-cast" Ni–Cr was mainly composed of dendritic pattern of gamma (γ) phase (austenite) as shown in Figure 10. After laser treatment, the austenite has re-crystallized forms of fine austenite grains as shown in Figure 11. Dispersed island of primary carbides in the alpha (α) (FCC) matrix is shown in Figure 12. The laser treatment has changed the dendritic structure to a uniform re-crystallised grain which is depicted in Figure 13.

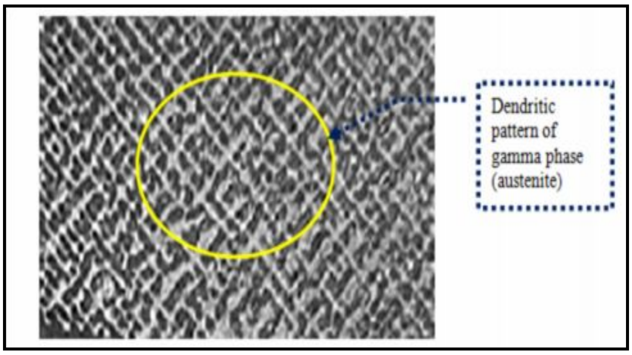


Figure 10. Microstructure of the test Ni–Cr base alloy as cast-dendritic pattern of gamma at 100×

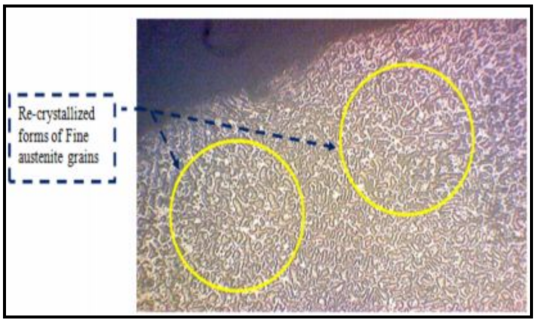


Figure 11 Microstructure of the test Ni –Cr base alloy after laser shock peening at the edge – 100X

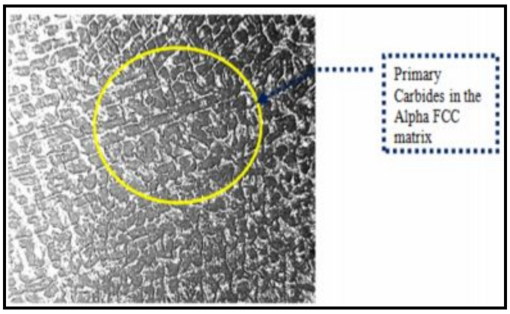


Figure 12 Micro Structure of the test Co-Cr base alloy as cast- Primary Carbides in the alpha matrix at 100X

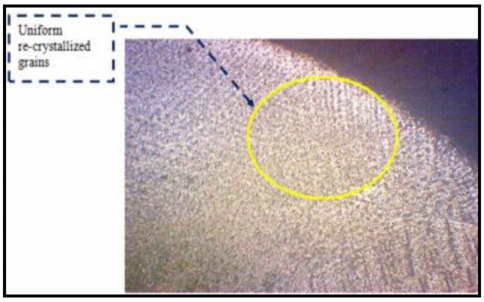


Figure 13 Microstructure of the Co –Cr alloy after LSP at the edge -100 x

SEM fractography analysis for fracture surfaces of tensile specimens of Ni- Cr alloys.

The SEM photomicrography and EDAX of irradiated (treated) region of fractured tensile specimen's surfaces were determined by Energy Dispersive Spectrometry (EDS). The fractured surface shows ductile appearance with sufficient plastic deformation. The high resolution image shows fine dimples the presence of crest and trough. No indefinable field was observed. The whole cross- section shows uniform fractured surface typical of a ductile failure as shown in figure 14 (a) and (b).

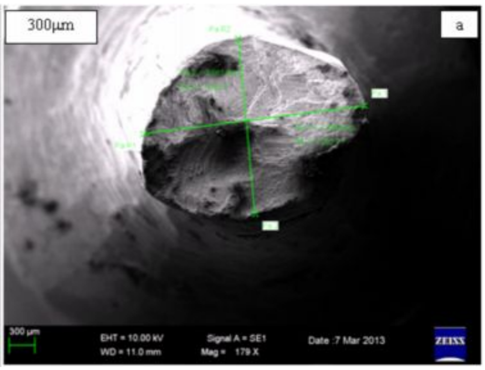


Figure 14 a. SEM micrograph of laser peened fractured surface of Ni-Cr alloy.

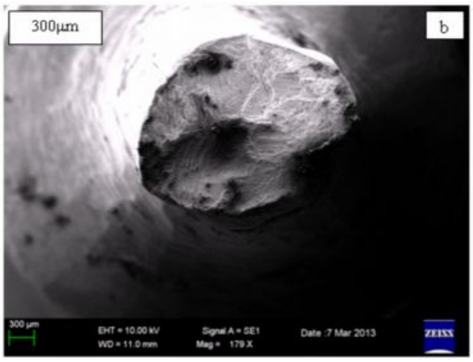


Figure 14 b. SEM Micrograph of Laser Peened fractured Surface Co – Cr alloys

The two images at the edge and core do not show much change in the microstructure of inter-dendrite pattern of grains as shown in figure 15 (a) and (b). The effect of laser treatment seen at left hand side edges of figure 15a. The SEM fractographic images for Ni –Cr alloy showed fine grained matrix which resulted in higher elongation due to crystallographic changes as shown in figure 15 (a). The depth of laser peening is up to 0.45 mm (450 microns). Nearly one third of the diameter is affected by the laser shock peening. The improved properties due to microstructure changes are evident.

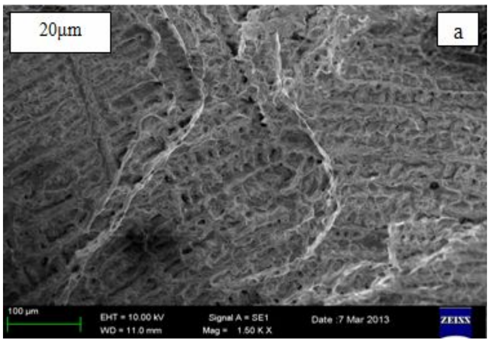


Figure 15 a. SEM micrograph of laser peened Ni-Cr specimen at the edge

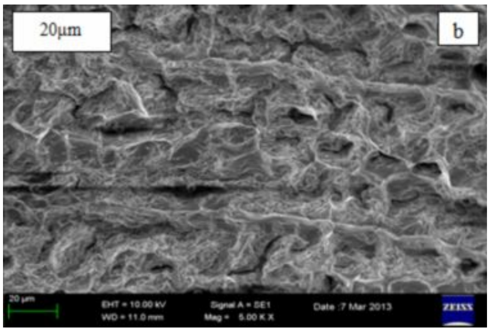


Figure 15 b SEM micrograph of laser peened Ni-Cr specimen at the core

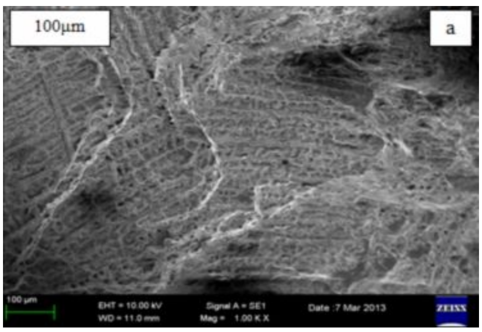


Figure 16 a SEM micrograph of laser peened fractured surface of tensile specimen of Ni –Cr Alloy

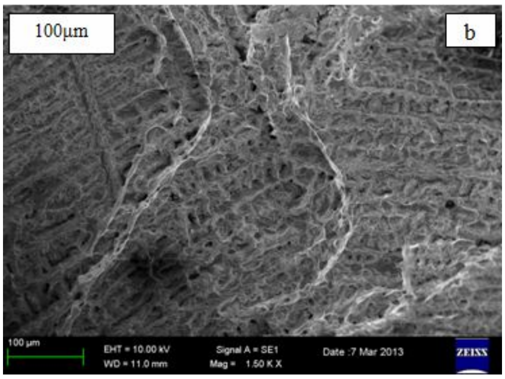


Figure 16 b SEM Micrograph of laser peened fractured surface of tensile specimen of Ni –Cr alloy at the core

Figure 16 (a) shows the edge of the tensile specimen with the re-crystallized austenite grains while the subsurface partially re-crystallized. Figure 16(b) shows the fractured surface at the core of Ni-Cr tensile specimen. The uniformity of "as cast" structure at the core shows the retention of primary dendrites retained and unaffected by the laser treatment.

3.3.3 SEM fractography analysis for fracture surfaces of tensile specimens of Co-Cr alloy.

The fractured surface of Co-Cr at the edge shows fine striations which have propagated to the core as shown in Figure 17(a). However at the core the microstructure is unaffected and shows the uniform dendrites as shown in Figure 17 (b).

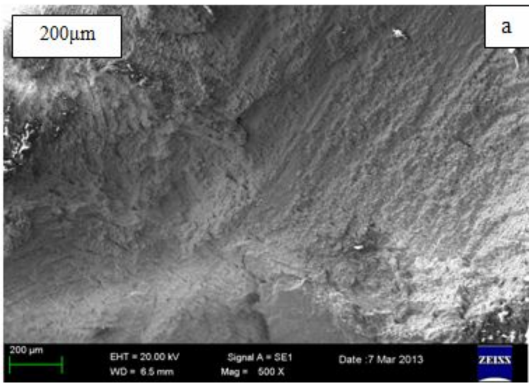


Figure 17 a SEM fractograph of LSP Co-Cr specimen at the edge – 500X

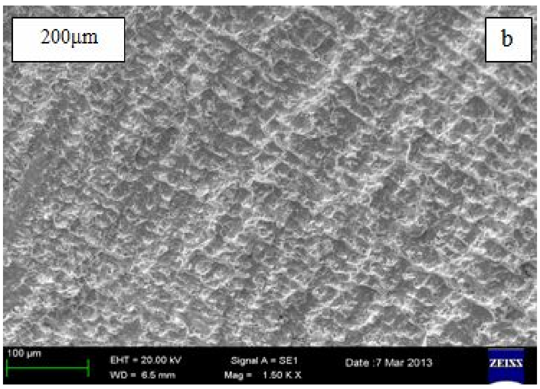


Figure 17 b SEM fractograph of LSP Co- Cr specimen at the core - 1.5Kx

3.3.4 Analysis of fractured surfaces of tensile specimens of Ni – Cr alloy and Co – Cr alloys

The EDAX value at the fractured surface of laser treated tensile specimen does not show wide variations of the constituent elements of Ni -Cr alloy as shown in figure 18 (a) and (b), figure 19(a) and (b).

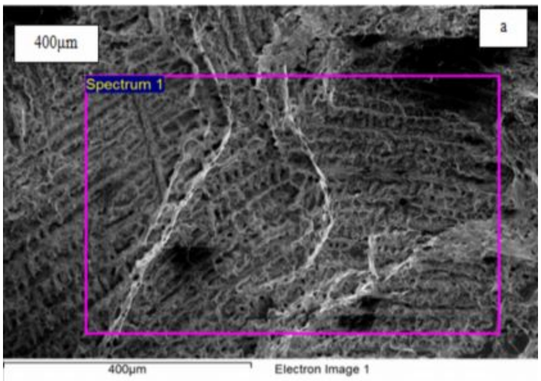


Figure 18 a EDAX: laser treated Ni-Cr elemental at spectrum Location

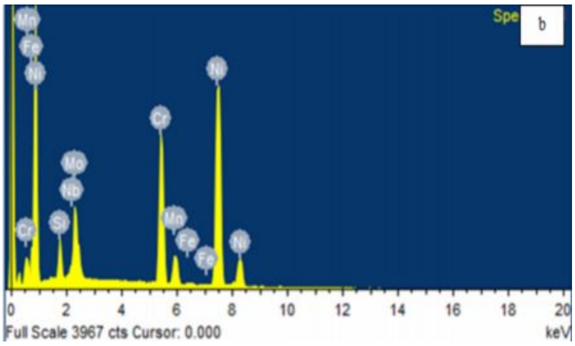


Figure 18 b EDAX: laser treated Ni-Cr composition of specimen

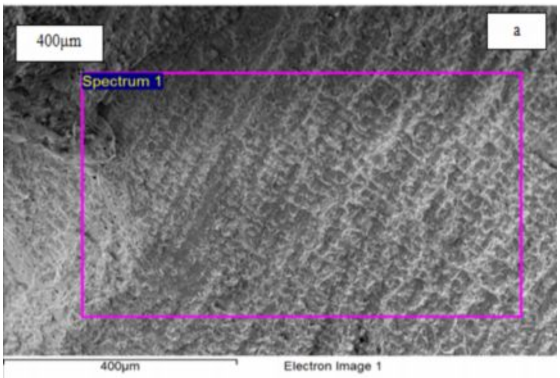


Figure 19 a EDAX: laser treated Co –Cr alloy fractured at Spectrum Location

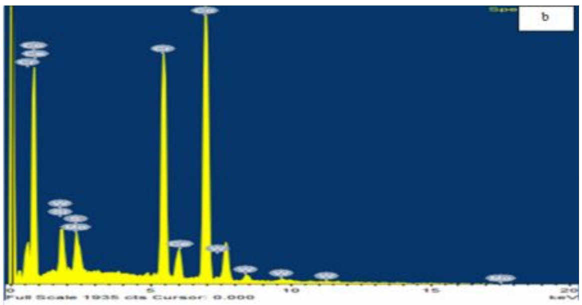
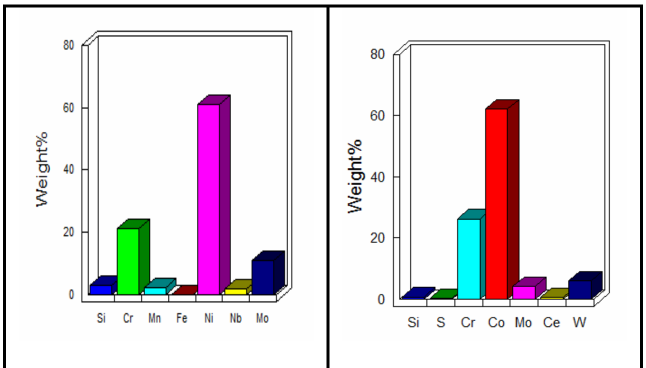


Figure 19 b EDAX: Elemental composition of tensile specimen of Co – Cr alloy after laser peening



(a) Ni- Cr alloy	(b) Co –Cr alloy

Figure 20 (a) and (b) Quantitative elemental results of fractured surfaces of tensile specimen of Ni–Cr and Co – Cr alloys after LSP.

The SEM with EDAX for Co-Cr shows some variations in composition probably due to alloy segregation. The results of the study showed significant differences in mechanical properties, surface hardness, and microstructure of the two selected base alloys finds place as alternate to the use of gold and titanium alloys. The application of the two selected non-noble base metals to prosthetic dental restorations seems to be more suitable.

Conclusions

The present study does not take into consideration of the allergies effect of Ni –Cr or the comparison of one over other. The effect of the Nd:YAG Laser Shock Peening (LSP) on the microstructure, micro hardness and mechanical behavior of Ni-Cr and Co-Cr alloys used for dental prostheses have been studied. The specific conclusions derived from the present study can be listed as follows**:**

- The laser shock peening of Ni – Cr and Co –Cr alloys has decreased the hard and brittle character of the alloys. This effect is induced by the laser treatment and the parameters used.

- The mechanical properties of these base alloys differed widely, and selection of the one over the other to be considered for their clinical application. Hence properties like mechanical physical and easy flexibility in manufacturing shall be taken into account. Ni-Cr fits better out of the two.

- The microstructure observations showed appreciable difference in morphology when treated at argon atmosphere. The improvement in percentage elongation up to 2 fold was observed for the Ni- Cr alloy specimens on treatment compared to untreated specimens. It is inferred that major clinical drawbacks can be removed by increasing workability. Co-Cr alloy on similar treatment has not yielded such properties that are workable.

- Difference in mechanical properties is directly related to their compositional differences, particularly secondary elements, such as Mn, Nb. These can form complex metallic phases to provide strengthening and microstructure refinement of grains to improve the mechanical properties and laser treatment on cast Ni-Cr and Co-Cr surfaces could produce reliable metal frameworks of prostheses and out of which Ni –Cr alloy has more preferred and makes it more fit to for dental applications**.**

Acknowledgements

Authors would like to express highest gratitude to Ragas Dental College & Hospital, A Unit of Jaya Educational Trust, Chennai and Mr.Saju Kuriakose, Director, M/s. Dentcare Dental Lab. Pvt. Ltd., Muvattupuzha, Kerala and M/s. Nanomaker's Laser at Trichy for providing facilities to conduct this study. Also the authors would like to thank the Faculty of Mechanical Engineering, Anna University, Chennai and T.R.Parthasarathy, Metallurgist, Metmech Engineers, Chennai for providing the facilities to carry out this study.

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