# Effect of heat treatment on the microstructure and hardness of Ni-Cr base metal alloys

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This paper describes the effect of heat treatment on nickel-chromium (hereafter referred to as Ni-Cr) base metal alloys to evaluate the following features: (a) The microstructure and hardness of Ni-Cr base metal alloys in as-cast and after heat treatment condition and (b) The effect of firing temperature on the metal substructure of the restoration for the changes in the mechanical and microstructural properties during porcelain firing.

Key words: Grain boundary, hardness, heat treatment, microstructure, Ni-Cr

## **INTRODUCTION**

The low price of nonprecious base metal alloys as compared to precious metals is a major attraction<sup>[1]</sup> and accounts for a sizeable portion of the fixed prosthesis alloys in the market.<sup>[2]</sup>

With regard to their physical properties, the increased strength and fusion properties of nonprecious alloys render them the preferred metal for more rigid, more thermally stable porcelain structures especially for multiple unit restoration.<sup>[3]</sup> These nonprecious alloys are used to improve casting and handling characteristics, porcelain bonding ability and corrosion resistance.<sup>[4]</sup> The esthetic advantage of an all porcelain labial margin combined with the strength of a metal substructure appear to be useful for the ideal ceramometal restoration. With the increased availability of nickel-based alloys than cobalt-based alloys, the focus of this study is on the nickel-chromium (hereafter referred to as Ni-Cr) alloys. These alloys undergo repeated porcelain firing at high temperature; hence, an understanding of the effect of firing on various properties of the metal substrate is important.

# MATERIALS AND METHODS

The samples for the study were fabricated using the conventional lost wax casting technique. The diameter of the samples was 6 mm and length, 12 mm. A wax pattern was produced using inlay wax sticks. The preformed sprue former (diameter, 2.5 mm and length, 25 mm) was attached horizontally to the wax pattern.

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The reservoir was attached to the sprue former 1 mm away from the pattern to prevent localized shrinkage porosity. The pattern was invested using Phosphate bonded investment (Whip mix, Corporation, USA) followed by burnout according to the manufacturer's instructions. After the burnout procedure, casting molds were transferred into the centrifugal electromagnetic induction-casting machine and casting procedure was carried out at 1420°C (casting temperature) for Ni-Cr (Wiron 99) alloy; Table 1 presents the various compositions. The castings were retrieved, sandblasted and sprues were cut.

Four Ni-Cr alloy rods (A, B, C, D) (diameter, 6 mm and length, 12 mm) were prepared and examined as as-cast and after heat treatment at 650°C, 750°C, 850°C and 950°C. These temperatures were selected as the average porcelain firing temperatures, i.e., 600°C to 950°C.

The microstructure and hardness of the four Wiron 99 rods were initially tested in the as-cast

Table 1: Composition of Ni-Cr alloys (Wiron 99)			
Nickel (Ni)	65%		
Chromium (Cr)	22.5%		
Molybdenum (Mo)	9.5%		
Silicon (Si)	1%		
Niobium (Nb)	1%		
Iron (Fe)	0.5%		
Cerium (Ce)	0.5%		
Carbon (C)	0.02%		
Aluminum (Al)	0%		
Cobalt (Co)	0%		

condition. Each of the four rods was further cut into 3 samples for testing at the 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> cycles of heat treatment. Heat treatment was carried out in an electric furnace for 1 h and cooled in air to room temperature.

The samples were polished with 1/0 to 4/0, grit emery abrasive paper polishing, disc polishing with fine alumina abrasive (0.05 µm).

The samples for metallographic examination were etched with a solution of saturated ferric chloride (100 g) dissolved in 25 ml of HCl for 5 s and observed under an optical microscope (Olympus metallurgical microscope with automatic camera) at 100×.

The hardness values of the samples were measured using Rockwell hardness tester equipped with diamondindenting tool, with an apex angle of 120° under a load of 150 kg ('c' scale)

#### RESULTS

The Ni-Cr alloy consisted of 16 samples cut from the 4 rod casts (labeled as A, B, C and D). Of the 16 samples, 4 were kept for as-cast study and remaining were subjected to the 1<sup>st</sup>, 2<sup>nd</sup>, 3<sup>rd</sup> heat treatment cycles at 650°C, 750°C, 850°C and 950°C. The as-cast and heat-treated conditions were compared. In heat-treated samples, microstructure showed a slight change in grain homogeneity. The Rockwell hardness values were not significantly altered by the heating cycle [Table 2].

Compared to the microstructure of as cast samples [Figure 1–4], the samples subjected to the 1<sup>st</sup> cycle of heat treatment showed cored or dendritic structure

Table 2: Hardness values in the cast and after heat treatment condition					
Rockwell hardness (Rc)	Sample (A) 650°C	Sample (B) 750°C	Sample (C) 850°C	Sample (D) 950°C	
Ni-Cr alloy	As cast 41 Rc	As cast 42 Rc	As cast 41 Rc	As cast 41 Rc	
	1 <sup>st</sup> cycle of heat treatment 41 Rc	1st cycle of heat treatment 38 Rc	1 <sup>st</sup> cycle of heat treatment 42 Rc	1 <sup>st</sup> cycle of heat treatment 40 Rc	
	2 <sup>nd</sup> cycle of heat treatment 39 Rc	2 <sup>nd</sup> cycle of heat treatment 37 Rc	2 <sup>nd</sup> cycle of heat treatment 40 Rc	2 <sup>nd</sup> cycle of heat treatment 38 Rc	
	3rd cycle of heat treatment 41 Rc	3rd cycle of heat treatment 40 Rc	3rd cycle of heat treatment 40 Rc	3rd cycle of heat treatment 40 Rc	



Figure 1: Microstructure of as-cast sample A

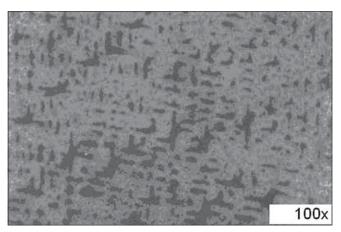


Figure 3: Microstructure of as-cast sample C

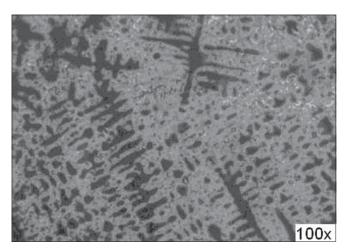


Figure 2: Microstructure of as-cast sample B

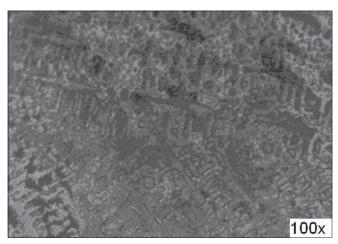


Figure 4: Microstructure of as-cast sample D

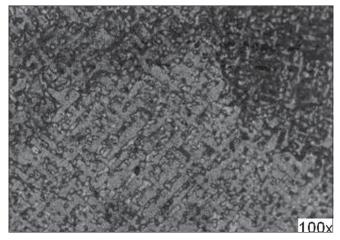


Figure 5: Microstructure after the 1st cycle of heat treatment (650°C)

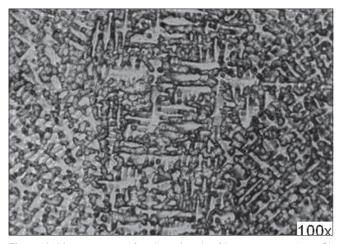


Figure 6: Microstructure after the 2<sup>nd</sup> cycle of heat treatment (650°C)

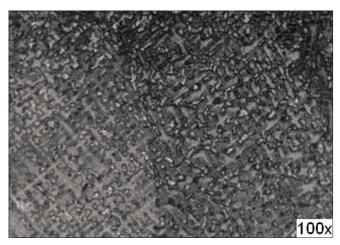


Figure 7: Microstructure after the 3<sup>rd</sup> cycle of heat treatment (650°C)

with primary and secondary arms. After the 1<sup>st</sup> cycle of heat treatment at 650°C, 750°C, 850°C and 950°C, few interdendritic precipitates and slight increase in grain homogeneity was observed [Figures 5, 8, 11 and 14]. In the 2<sup>nd</sup> cycle of heat treatment, the amount of intradendrite precipitation slightly increased and

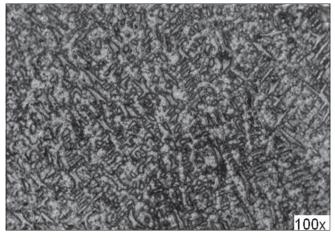


Figure 8: Microstructure after the 1st cycle of heat treatment (750°C)



Figure 9: Microstructure after the 2<sup>nd</sup> cycle of heat treatment (750°C)

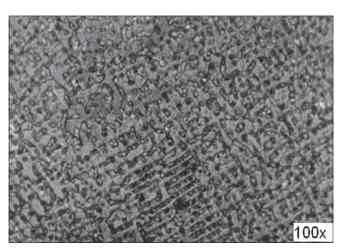


Figure 10: Microstructure after the 3<sup>rd</sup> cycle of heat treatment (750°C)

more grain homogeneity was observed [Figures 6, 9, 12 and 15]. In the 3<sup>rd</sup> heat treatment cycle, further dendritic precipitation and grain boundary homogeneity was observed [Figures 7, 10, 13 and 16]. This pattern of interdendritic precipitation and homogeneity in

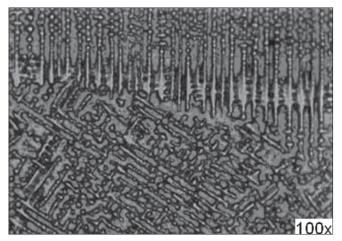


Figure 11: Microstructure after the  $1^{st}$  cycle of heat treatment (850°C)

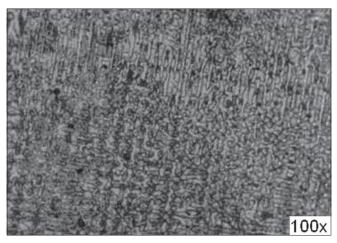


Figure 14: Microstructure after the  $1^{st}$  cycle of heat treatment (950°C)

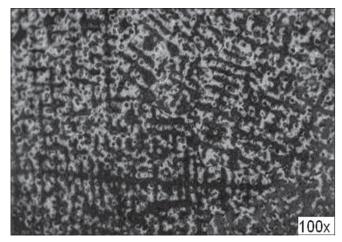


Figure 12: Microstructure after the 2<sup>nd</sup> cycle of heat treatment (850°C)

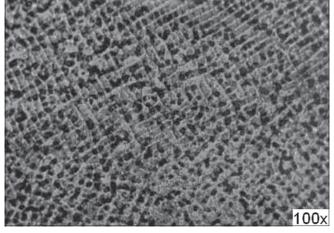


Figure 15: Microstructure after the  $2^{nd}$  cycle of heat treatment (950°C)

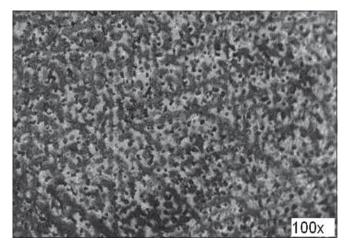


Figure 13: Microstructure after the  $3^{rd}$  cycle of heat treatment (850°C)

grain boundaries were observed in all the samples subjected to heat treatment cycles at 650°C, 750°C, 850°C and 950°C.

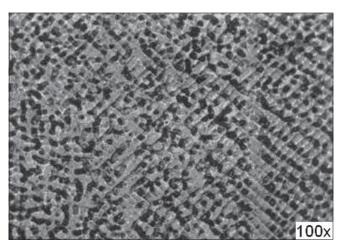


Figure 16: Microstructure after the  $3^{rd}$  cycle of heat treatment (950°C)

## DISCUSSION

## The use of Ni-Cr alloys continues to be an important

aspect of dental restorative materials. The present study was designed to evaluate the microstructure and hardness of Ni-Cr in as-cast and after heat treatment on the metal substructure of the restoration with regard to the changes in mechanical and microstructural properties. In the present study, the Ni-Cr alloy showed slight change in grain homogeneity of the microstructure where the alterations in the hardness values were not significant. These results are in conformation with the previous studies by Morris et al,<sup>[5]</sup> Boyadjain et al<sup>[6]</sup> and Chew et al.<sup>[7]</sup> After each heat treatment, the microstructural analysis revealed a slight increase in the amount of interdendritic precipitation and grain homogeneity. This is similar to the observation of Olivieri et al.<sup>[8]</sup> The properties of microstructure and hardness are not the only parameters determining clinical success. The interaction between these and other properties, such as tensile strength, yield strength, percent elongation, tarnish/ corrosion resistance, porcelain to metal compatibility and biocompatibility, should also be considered while judging the clinical success, which are not included in the present study.

## CONCLUSION

Within the limitations of this work, the following could be concluded:

- At different heat treatment temperatures of 650°C, 750°C, 850°C and 950°C, Ni-Cr alloy showed a slight change in grain homogeneity in the microstructure.
- The hardness values did not significantly change because of the heat treatment.

- No significant effect on the metal substructure could be predicted with regard to the microstructure and hardness while firing porcelain.
- The use of elevated temperatures above that recommended by the manufacturers could lead to grain growth, decrease in strength, fissures, cracks, etc.

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