Effect of PFM Firing Cycles on the Mechanical Properties, Phase Composition, and Microstructure of Nickel-Chromium Alloy

Mohd. Anwar, BDS,1 Arvind Tripathi, MDS, FACS, MNAMS, FICD,2 Sushil Kumar Kar, MDS,1 & K. Chandra Sekhar, MTech3

1Department of Prosthodontics, Saraswati Dental College and Hospital, Lucknow, India
2Dean Postgraduate Studies and Research, Saraswati Dental College and Hospital, Lucknow, India
3Department of Materials Science and Engineering, Indian Institute of Technology, Kanpur, India

Keywords
Porcelain fused to metal; optical microscopy; diffraction; tensile testing; base metal alloys; casting alloys.

Correspondence
Arvind Tripathi, MD 10, Sector C, Aliganj, Lucknow-226024, Uttar Pradesh, India.
E-mail: atrip2006@gmail.com

The authors deny any conflicts of interest.

Accepted February 2, 2015

doi: 10.1111/jopr.12328

Abstract

Purpose: The purpose of this study was to compare the mechanical properties of beryllium-free nickel-chromium (Ni-Cr) dental casting alloy before and after each porcelain firing cycle (once fired, twice fired, and thrice fired) and to relate these properties to the microstructural changes and changes in X-ray diffraction patterns of Ni-Cr alloy that occur after each porcelain firing cycle.

Material and methods: Forty tensile bar specimens and 20 disc-shaped specimens of Ni-Cr alloy were prepared. These specimens were divided into four groups. The first group was not heat treated and tested in the as-cast condition, thus serving as control group. The second, third, and fourth groups were fired once, twice, and thrice, respectively. Tensile bar specimens were loaded to failure in tension using a universal testing machine. Values of ultimate tensile strength, 0.1% yield strength, and percentage elongations were determined. Microstructural study and hardness testing were done using an optical microscope and digital Vickers hardness tester, respectively, on disc-shaped specimens. Disc-shaped specimens were again used to obtain the X-ray diffraction patterns by using diffractometer Bruker D8 focus. Statistical comparisons of the mechanical properties and hardness of the alloy were made with ANOVA. Intergroup comparisons of the data in the as-cast and fired specimens were analyzed by applying Tukey’s HSD multiple comparison tests.

Results: Before porcelain firing, the alloy exhibited higher ultimate tensile strength (548 MPa), 0.1% yield strength (327 MPa), hardness (192 HV), and lower elongation values (18%). After each firing cycle, there was a significant (p < 0.001) decrease in ultimate strength (464 MPa for three times fired specimens), 0.1% yield strength (284 MPa for three times fired group), and hardness (164 HV for three times fired group) and significant (p < 0.001) increase in elongation value (28% for three times fired group) of Ni-Cr alloy. The microstructure of the control group specimen exhibited heterogeneous microstructure, and after each firing, microstructure of the alloy was gradually homogenized by formation of grain boundaries at the interdendritic interfaces. X-ray diffraction pattern shows that the alloy exhibited four strong diffraction peaks within the range of 2θ = 40° to 100°. After a third firing, intensity of these planes increased.

Conclusions: Results of this study confirmed that nickel-based alloys become weaker after each firing process. After firing treatment, the microstructure of alloys showed decreased dendritic structure (i.e., homogenization, which was responsible for decrease in strength and an increase in ductility of the alloy); however, this decreased strength and hardness of Ni-Cr alloy after heat treatment was still superior to those of the most noble metal alloys used in dentistry. X-ray diffraction study showed that firing process led to relieving of stresses, which ultimately resulted in stability in the crystal structure of alloy.
Porcelain has been used for fabricating esthetic dental restorations since the early 1900s. The first published report describing the successful use of porcelain-fused-to-alloys was published in the mid-1950s. Since then, research and improvements in materials and techniques have dramatically increased the use of metal-ceramic restorations.

To fabricate a metal-ceramic restoration, a metal substructure is waxed, cast, finished, and heat-treated (oxidized). A thin layer of opaque porcelain is fused to the oxidized metal surface to establish a porcelain-metal bond and hide the color of the substructure. Then dentin and enamel porcelains, sometimes referred to as body and incisal porcelains, respectively, are fused to the opaque porcelain, shaped, stained to improve the esthetic appearance, and finally glazed.

The porcelain-fused-to-metal (PFM) firing process is conducted in a vacuum in three to four stages at temperatures ranging from 950 to 1010°C. Often, only the facial aspect of the restoration is veneered, leaving the lingual, palatal, occlusal, and subgingival margins exposed. Although considerable functional stress is borne by the ceramic portion of a metal-ceramic restoration, success of the entire prosthesis depends largely on the mechanical properties of the metal substructure.

The most frequently used alloys for metal-ceramic restorations are nickel-based alloys. The major constituents of nickel alloys are nickel and chromium; however, they also contain a wide range of minor alloying metals.

Extensive solid solubility of chromium was found in nickel in the binary phase diagram of the nickel-chromium (Ni-Cr) system. At room temperature, nearly 37 wt% Cr remains dissolved in a matrix called “gamma.” Other elements were also added to the alloy to enhance its properties. Chromium imparts corrosion resistance and solid solution hardening to some extent, while other additions would improve the solid solution hardening or precipitate formation.

High-temperature heat treatments and repeated PFM firing cycles have been shown to affect alloy microstructures, surface oxides, corrosion, and physical and mechanical properties. To date, few studies have evaluated the effects of high temperature attained during PFM firing on the mechanical properties and microstructure of Ni-Cr alloy. However, no study has been carried out to evaluate the changes in mechanical properties, microstructure, and X-ray diffraction (XRD) pattern of the alloy after each firing. To keep the effect of heat treatment on the Ni-Cr alloy minimal, it is important to evaluate the effects of PFM firing cycles on the properties of Ni-Cr alloy.

Therefore, the purpose of this study was to compare the mechanical properties of Ni-Cr dental casting alloy before and after each porcelain firing cycle (once fired, twice fired, and thrice fired) and to relate these properties to the microstructural changes and changes in XRD patterns of Ni-Cr alloy that occur after each porcelain firing cycle.

Materials and methods

An Ni-Cr alloy (Bellabond plus- Lot no. 733520413; Bego Goldschlägerei Wilh. Herbst GmbH & Co., Bremen, Germany) was selected for this study. The manufacturer states (in the users catalog) that the composition of the alloy is 65.2% nickel, 22.5% chromium, 9.5% molybdenum, and iron, silicon, manganese, and niobium in traces.

Two types of specimens were prepared for the study: tensile bar and disc-shaped specimens. Uniform tensile bar specimens were prepared by injecting molten wax into a specially designed split metal mold with dimensions recommended by the American Dental Association specification no. 14 for tensile strength testing of dental base metal casting alloys. Disc-shaped specimens with dimensions of 5 mm in thickness and 8 mm in diameter were prepared by injecting molten wax into a custom-made mold within the putty poly(vinyl siloxane) impression material.

Forty tensile bars were cast according to the study design. The cast bars were randomly placed in four groups of 10 each. Similarly, 20 disc-shaped specimens were cast and placed in four groups of five each. Firing sequences employed in the fabrication of metal-ceramic restorations were also used for firing of specimens in this study, but without the application of porcelain.

Figure 1 (A) Tensile bar specimens. (B) Disc-shaped specimens mounted in acrylic molds.

The first group (group C) specimens were not heat-treated and tested in the as-cast condition, and thus served as the control group. The second group (group 1) was fired once (opaque fire) and air-cooled, the third group (group 2) was fired twice (opaque and body fire) and air-cooled, and the fourth group (group 3) was fired thrice (opaque, body, and glaze fire) and air-cooled.

Mechanical properties

The ultimate tensile strength is defined as the maximum stress a material can withstand before fracture. The clinical success of metal-ceramic restorations depends upon the mechanical properties of both materials. Leone and Fairhurst have shown that the tensile strength of porcelain is relatively low (i.e., 37 MPa). When a metal-ceramic structure bends, destructive tensile stresses develop within the porcelain veneer. Hence, the metal substructure should possess sufficient strength properties and should follow ideal structural design considerations, which contribute to the rigidity and resistance to permanent deformation.

Clinically, yield strength represents the level of stress below which a restoration will behave elastically and above which
a restoration will permanently deform and fail.\textsuperscript{18} Although a sufficiently high value of yield strength is essential for a ceramic alloy, too-high values may create difficulties when the casting is adjusted in the dental laboratory.\textsuperscript{21}

Casting alloys used for porcelain-fused prostheses exhibit percentage elongations (which represents the quantitative value of ductility) greater than 10\%. As described by Moon and Modjeski\textsuperscript{22} while considering the ease of adjustment for cast restorations, the practitioner must remember that both properties (i.e., yield strength and percentage elongation) are involved. Alloys with high yield strength cannot be burnished manually, even if they have high values of percentage elongation.\textsuperscript{21}

Tensile bars were used for the testing of ultimate tensile strength, 0.1\% offset yield strength, and percentage elongation. Engineering stress was used to calculate the strength values. A fully computerized EZ50 material testing machine (Lloyd Instruments-Ametek, Inc. West Sussex, UK) was used to determine the mechanical properties.

Before starting the test, the gauge length of the tensile bar specimen was measured by digital vernier caliper, and two marks were placed to define the gauge length. Later, these marks were used for the calculation of percentage elongation after rupture. Each tensile bar specimen was subjected to tensile test in this machine with a 0.5 mm/min crosshead speed\textsuperscript{6} and a 50 kN load cell.

Once the machine started, it began to apply an increasing load on the specimen. Thus, the specimen tended to elongate with respect to time upon pulling speed (0.5 mm/min) and finally broke after some time. Throughout the test, the control system and its associated data analysis and material testing software (Nexygen Plus 3; Lloyd Instruments Ltd.) recorded the load and extension of specimen. Following rupture of the test bar, the ultimate tensile strength was computed by dividing the maximum recorded load by the diameter of tensile bar.\textsuperscript{17} An offset of 0.1\% was used as an arbitrary value to calculate the yield strength. This was done by plotting a line to represent the offset parallel to the straight line portion of the stress-strain curve, which was executed by computer software designed for the testing machine.\textsuperscript{17} The percentage elongation was calculated by placing the fractured specimen halves together and measuring the new separation between the original gauge length marks.\textsuperscript{23}

### Microstructural study

Disc-shaped specimens were prepared metallographically for microstructural study. Specimens were first mounted in cylindrical molds using autopolymerizing acrylic resin (Fig 1B). Gross grinding of the specimens was done by a belt grinder. Finer grinding of the specimens was done with silicon carbide abrasive papers (Buehler, Lake Bluff, IL) with the help of a grinder-polisher machine (Beta; Buehler).\textsuperscript{24} The typical sequence of grinding began with 120- or 180-grit papers and proceeded through 240, 320, 400, 600, 800, 1000, and finally 1200 grit. Specimens were wet polished with 1 and 0.05 \( \mu \)m aluminium oxide particles on metallographic polishing tables, cleaned with distilled water, and finally air-dried. Specimens were etched for 20 seconds using a mixture of 20 vol\% HNO\textsubscript{3} (nitric acid) and 80 vol\% HCl (hydrochloric acid).\textsuperscript{3} For viewing the microstructures of etched specimens, an optical microscope (Leica DM 6000M; Leica Microsystems Ltd., Mannheim, Germany) coupled with a digital image capturing device and software (Leica Application Suite) were used. Photomicrographs were taken at 50\( \times \), 200\( \times \), and 500\( \times \) magnifications.

### Hardness testing

Vickers hardness number (VHN) is generally measured for dental alloys. Though hardness is an important physical property, high values of hardness cause difficulty in trimming and finishing castings in the lab. Alloys with VHN values exceeding that of enamel (approximately 350) cause abrasive wear of opposing teeth.\textsuperscript{21} The same mounted disc specimens used for microscopic examination were again polished as described earlier and used for hardness testing.

Vickers microhardness testing was done by a digital display Vickers hardness tester (model HVS-50: Banbros Engineering Pvt. Ltd. Delhi, India) using a 136\(^\circ\) diamond pyramidal indenter with a 1-kgf load and 15-second dwell time. Tests were executed under a microscope supplied with the device for microhardness test. The hardness of the specimen was determined automatically using a digitized microhardness tester by measuring the distance between the diagonals of the indentation. Four random determinations were made for each of the mounted specimens, and the mean values were calculated for statistical purposes.\textsuperscript{6}
X-ray diffraction study

XRD is used for characterization of the alloy and to obtain bulk information on metallurgical properties and phase composition before and after each firing cycle. Disc-shaped specimens from each group were again polished and used for XRD study.

XRD patterns were conducted at room temperature over a range of 40° to 100° using a diffractometer (D8 Focus; Bruker, Madison, WI) equipped with an Ni filter, and calibrated using aluminum standard. The diffractometer was operated with Cu kα radiation at 0.02 step size. Features on the XRD pattern were identified using DiffracPlus X-ray diffraction software (Bruker).

Statistical analysis

The data obtained from tensile and hardness testing were subjected to statistical analysis. Means of the data of as-cast and fired specimens were analyzed for differences with ANOVA. Intergroup comparison of the data in as-cast and fired specimens was analyzed by applying Tukey’s HSD multiple comparison tests.

Results

Mechanical properties

Mean and standard deviation values of ultimate tensile strength, 0.1% yield strength, percentage elongation, and Vickers hardness of all the groups are listed in Table 2. Alloy specimens in the control group exhibited the highest strengths among the groups. After first firing (group 1), the ultimate tensile strength and 0.1% yield strength was reduced to 525 and 319 MPa, respectively, and after the second firing, the respective strengths were reduced to 498 and 304 MPa. Finally, after the third firing (group 3), ultimate tensile strength and 0.1% yield strength were further reduced to 464 and 284 MPa, respectively.

Elongation of the alloy increased from 18% for the control group to 19% for group 1 and 23% for group 2. The highest elongation was recorded for group 3 alloy specimens.

Average Vickers hardness values ranged from 164 to 192. Control specimens showed the highest hardness value, while group 3 specimens showed the lowest values. The values in groups 1 and 2 were of middle order.

ANOVA revealed statistically significant intergroup difference (p < 0.001) in ultimate tensile strength, 0.1% yield strength, percentage elongation, and hardness. Each group had a characteristic value for its properties, which varied in a specific range only.

On subjecting the data of ultimate tensile strength, 0.1% yield strength, percentage elongation, and hardness to further post-hoc assessment using Tukey’s HSD test for intergroup comparison, the maximum difference was observed between groups C and 3 and minimum difference between groups C and 1. Statistically, differences among the groups were significant at a 95% significance level (p < 0.001).

Microstructural study

Microstructure of the control group specimen exhibited extensive solid solution of γ (gamma) matrix in the form of dendrites. Darker interdendritic areas (black arrows in Figs 2A-C) were present in continuation with the white dendritic regions (white arrow in Figs 2A-C). Grain boundaries were not evident.

The microstructure of group 1 (once-fired specimens) showed partial coarsening of the dendrites as compared to the microstructure of group C specimens. Dendrites contained a primary arm (black arrow in Figs 2D and E) and a secondary arm (white arrow in Figs 2D and E), lying perpendicular to each other, but grain boundaries were not clear.

The microstructure of group 2 (twice-fired specimens) started to partially reveal the grain boundaries at interdendritic interfaces (white arrow in Fig 2G). At higher magnifications, formation of random grain boundaries was clearly evident (white arrow in Figs 2H and I). Some precipitates were seen in the dendritic region (black arrows in Fig 2D), and dendrites became coarser due to the effect of further heat treatment.

The microstructure of group 3 specimens showed decomposition of dendritic structure by dissolving its arms and topography of the matrix (white arrow in Figs 2J-L). Defined grain boundaries started to form at the interfaces of dendrites, along with formation of triple-point junction at the edges (black arrow in Figs 2K and L).

X-ray diffraction study

Representative XRD diffraction patterns for the as-cast (control) and once-fired, twice-fired, and thrice-fired specimens are presented in Figure 3. The XRD results of Ni-Cr alloy were indexed. The alloy was a primarily face-centered cubic (FCC) solid solution. In all cases, consistent results were obtained before and after firing. The peaks obtained at 2θ angle of approximately 42°, 50°, 73°, and 89° (if present in the given pattern) were related to (111), (200), (220), and (311) planes, respectively. Alloys exhibited four strong diffraction peaks within the range of 2θ = 40° to 100°. After the third firing, the intensity of these planes relatively increased.

Discussion

Firing of porcelain on metal substructure produces changes in the microstructure, mechanical properties, and XRD pattern of the alloy that could influence the behavior of an alloy and its clinical efficacy during long-term use. On the basis of the results obtained from the present study, a general consensus has been drawn regarding the strengths, hardness, and percentage elongations of one commercial Ni-Cr alloy before and after repeated porcelain firing cycles. Before porcelain firing, the alloy exhibited higher strength and hardness and lower elongation values, and after each firing cycle, there was a gradual decrease in strength and hardness and a gradual increase in percentage elongation value of the Ni-Cr alloy. These results are in agreement with those of the previous studies carried out by Moffa et al.,9 Morris,7 Huget et al.,9 Chew et al.,10 and Qiu et al.23 Results of the present study are also similar to those observed by Winkler et al.8 (they used an Ni-Cr base metal alloy Ticon; Ticonium Co., Albany, NY); however, in context of the effect of heat treatment on hardness of Ni-Cr alloy, their findings are in contrast to those of the present study (hardness increased from 357 to 387 HV after five simulated porcelain firing cycles).
Table 2 Statistical analysis of mechanical properties

<table>
<thead>
<tr>
<th>Groups*</th>
<th>Ultimate tensile strength (MPa) Mean ± SD</th>
<th>Yield strength (0.1% offset) (MPa) Mean ± SD</th>
<th>Elongation (%) Mean ± SD</th>
<th>Hardness (HV) Mean ± SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control group</td>
<td>548 ± 6</td>
<td>327 ± 6</td>
<td>18 ± 1</td>
<td>192 ± 1</td>
</tr>
<tr>
<td>Group 1</td>
<td>525 ± 8</td>
<td>319 ± 6</td>
<td>19 ± 1</td>
<td>181 ± 1</td>
</tr>
<tr>
<td>Group 2</td>
<td>498 ± 7</td>
<td>304 ± 5</td>
<td>23 ± 2</td>
<td>175 ± 2</td>
</tr>
<tr>
<td>Group 3</td>
<td>464 ± 9</td>
<td>284 ± 7</td>
<td>28 ± 1</td>
<td>164 ± 1</td>
</tr>
</tbody>
</table>

*All the intergroup differences were significant at 95% significance level.

Figure 2 Representative optical photomicrographs of control group (A, B, C), group 1 (D, E, F), group 2 (G, H, I), and group 3 (J, K, L) specimens. Black bar in from left to right represents 500, 100, and 50 μm, respectively. Black arrows in (A, B, C), (D, E), (I), and (J, K, L) represent interdendritic region, primary arm, precipitates, and dissolution of matrix, respectively. White arrows in (A, B, C), (D, E), (G, H, I), and (K, L) represent dendrites, secondary arm, grain boundaries, and formation of triple point junction, respectively.

Hardness results are in contrast to the findings of Lin et al., who found that the hardness of Litecast alloy (a non-Be containing Ni-Cr alloy) increased significantly ($p < 0.01$) after PFM firing (before firing: 138 HV, after PFM firing: 149 HV). The Ni-Cr alloy used in their study had 15% molybdenum content without titanium and niobium additions. The molybdenum content in the present study was 9.5% with niobium addition (molybdenum imparts solid solution strengthening, and niobium reduces the melting temperature of the alloy). So, differences in hardness value after PFM firing might be related to the compositional differences.
Since the late 1970s and early 1980s, base metal alloys have been used exclusively as they were a cost-effective and superior alternative to noble metal alloys. ADA specification no. 5 gave a range for mechanical properties of cast gold alloy. According to this specification, yield strength, elongation, and hardness of gold alloy range from 80 to 270 MPa, 10% to 39%, and 60 to 150 HV, respectively. For base metal alloys, the requirements in ultimate tensile strength, yield strength, elongation, and hardness are 539 to 919 MPa, 180 to 858 MPa, 10% to 28%, and 175 to 360 HV, respectively. When noble metals and base metal alloys were compared for mechanical properties, the mechanical properties of base metal alloys were still superior to those of noble metal alloys, even after the third firing, and this decreased strength is still sufficient for the longevity of restorations.

The highly “cored” or dendritic microstructure of the as-cast specimen in the present study is similar to that reported by several authors, including Qiu et al.\textsuperscript{23} and Pimenta et al.\textsuperscript{12} who analyzed alloys with composition similar to this study. The dendritic microstructure of the control specimen is considered to be heterogeneous in nature, and this heterogeneous structure is ultimately responsible for the higher strength and hardness and lower elongation values obtained in these specimens.

Group 1 specimens showed no significant differences in the microstructure from those of the control group. So, after one firing, the heat treatment did not significantly affect the microstructure of the alloy. The transition in the microstructure was observed in mechanical properties as a slight decrease in tensile properties and hardness, with a slight increase in elongation value of the alloy.
In the microstructure of group 2 specimens, some precipitates, which were probably constituents of chromium, molybdenum, niobium, and silicon carbides, were also observed in the dendritic region. The microstructure in this group also started to reveal the grain boundaries partially. It can be understood that the heterogeneity of the dendritic structure gradually relieved stresses to become homogenized. The response of the microstructural changes is also seen in the mechanical properties by a decrease in mean ultimate tensile strength from 548 to 498 MPa and a decrease in hardness from 192 to 175 HV with an increase in percentage elongation from 18 to 23.

Some degree of homogeneity was observed in the microstructure of group 3 specimens by the formation of defined grain boundaries along with formation of triple-point junctions, thereby indicating that the dendrites are stress relieved due to the effect of repeated firing cycles; however, the thicker and irregular nature of grain boundaries in the microstructure of this group showed that porcelain firing (i.e., heat treatment) did not completely homogenize the microstructure of the alloy. Granular precipitates and less apparent dendrites were observed at the grain boundaries. Therefore, it can be confirmed that the matrix of the Ni-Cr alloy did not change by firing procedures except stress relieving in the material. This was observed in mechanical properties as a rapid decrease in strength and hardness and greater elongation values of the alloy in three times fired groups.

Indexing of XRD patterns showed that the pattern is mostly related to a major element in the composition; however, alloying elements may shift the pattern. For Ni-Cr alloy, the peaks reflected a nickel solid solution with an FCC crystal structure. Addition of a number of substitute elements like molybdenum in Ni-Cr alloy decreases the lattice parameters by substitution of Ni. Diffraction takes place at lower angles in such substitutional solid solution crystalline lattices as compared to nickel’s standard pattern.

No significant degree of peaks existed except the γ phase of Ni-Cr alloy, ruling out the presence of additional phases. The diffraction patterns of control group specimens and those fired once had γ phase peaks of (111), (200), and (220), indicating that athermal martensitic transformation did not occur during the solidification process when the γ phase was stacked. The γ (200) peak became sharper and more intense after specimens were subjected to different firing cycles. This indicated that the γ phase was stabilized by the firing process temperature and retained at room temperature, suggesting that the general athermal martensitic transformation from γ to ε phase might be suppressed or undetectable. Accordingly, further firing cycles suppressed the lattice strains, leading to an increase in volume fraction of γ phase and grain growth at room temperature. However, further study is required to explain the relevant mechanism of the suppression of athermal transformation.

When XRD data were analyzed in relation to optical microscopy, a discrepancy appeared to arise, since only solid solution was detected by XRD; however, any precipitates believed to be observed in the photomicrographs mentioned earlier were perhaps not in enough quantity to be detected with XRD parameters used in the present study. By these results of XRD analysis, it can be concluded that the matrix was still FCC up to the third firing, and no precipitation had taken place during the firing cycles, which was confirmed by the absence of any additional peaks in the XRD pattern other than that described for the Ni-Cr alloy.

The lattice strains were almost reduced, which can be understood by high-intensity diffraction peaks of the third-group specimens as compared to the first group. This indicates that the microstructure was stress relieved, and stability was obtained in the crystal structure. The stabilized structure was responsible for reduction in ultimate tensile strength, yield strength, and hardness value, and contributed to an increase in ductility of the alloy.

In the present study, variables such as ultimate tensile strength, yield strength, hardness, and percent elongation of the alloy were used to judge the clinical success of PFM restorations; however, some important properties such as corrosion resistance, metal ion release, oxide layer formation, effect of different casting conditions, and porcelain-metal compatibility were not considered. These parameters should also be taken into account in future studies to determine the clinical success of the PFM restorations.

Conclusions

The results of the present study confirmed that nickel-based alloy becomes weak after heat treatment and exhibits a decrease in ultimate tensile strength, yield strength, and hardness and shows an increase in percentage elongation. On the basis of these findings and within the limitations of the study, it may be concluded that:

1. In the as-cast condition, the microstructure of alloy specimens exhibited a dendritic structure (i.e., nonhomogenous structure), which was responsible for the higher strength (mean ultimate tensile strength = 548 MPa; 0.1% yield strength = 327 MPa), hardness (192 HV), and lower elongation value (18%) of the alloy.
2. After heat treatment, microstructures of the alloy showed decreased dendritic structure (i.e., homogenization), which led to grain growth. Hence, an improvement in ductility (28%) and concurrent decrease in strength (mean ultimate tensile strength = 464 MPa; 0.1% yield strength = 284 MPa) was observed.
3. The XRD study showed that the firing process led to relieving of stresses, which ultimately resulted in stability in the crystal structure of the alloy.

Acknowledgment

The authors sincerely thank the technical staff of the Department of Materials Science and Engineering, Indian Institute of Technology, Kanpur, India, for their help and support in the study.

References