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Bulk Recycling of Ni-Cr-Mo Dental Alloy - A Sustainable Approach

Sumeet Batra[1], Xitij Detroja[1], Akash Biradar[1], Jayaprakash K[2], Nada Z. Mohammed[3] and Rijesh. M[1*]

[1]*Department of Manufacturing Engineering, School of Mechanical Engineering, Vellore Institute of Technology, Vellore, Tamilnadu-632014, India*

[2]*Department of Dental Materials, Biomaterials and Research Center, Yenepoya Dental College, Yenepoya University, Mangalore, Karnataka-575018, India*

[3]*College of Dentistry, University of Mosul, Mosul- 41001, Iraq*

Many dental alloy manufacturers instruct not to recast alloys, but the dental laboratories reuse the casting surplus for economic reasons. It is a controversial topic in dental practice, so the present study attempts to assess the effects of recasting Ni-Cr-Mo alloys. Three sets of alloy pellets were recast. The first set was melted and allowed to solidify. The second and third sets were recast two times and three times, respectively. The elemental composition of all the recast samples was analysed using ED-XRF (Energy Dispersive X-ray Fluorescence) spectrometer. The variation in the chemical composition for the number of recasting was reported. It was observed that the recasting for one time doesn't change the elemental composition to a considerable extent. However, further increase in the number of recasting two and three times, the depletion of the major alloying elements (Ni, Cr, and Mo) was notable. Hence, the current study intends to calculate the exact amount of the depleted elements after each recast. Also, to have a theoretical analysis of the elements necessary for making a master alloy that can be added during the recasting to avoid changes in elemental composition. Moreover, the microstructure of the recast samples was observed using an optical electron microscope (OEM). No drastic variation in the microstructure was observed for the alloy with several melting and solidifying cycles, except for the orientation of the dendritic arm. Furthermore, to confirm the mechanical strength of the recast alloy Vickers microhardness test was conducted. The average microhardness of the base Ni-Cr-Mo alloy was 216 HV, and recasting once does not affect the hardness value. However, the three-time recast alloy showed only a 9% decrement. Finally, it can be concluded that the number of recasting can be as many times provided depleted elements are added in exact proportion after each recast. The current research suggests recycling dental alloys in bulk outside a dental clinic, and a dentist should responsibly segregate different base metal alloys and promote sustainable dentistry.

Keywords: Ni-Cr-Mo alloys; Recast; Chemical composition; Microstructure; Hardness

I. INTRODUCTION

Dental practices generate notable amounts of waste, including recyclable materials, domestic other than food, hygiene, clinical, hazardous, and food wastes (Duane *et al.,* 2019). It is the legal and professional responsibility of the dental team to ensure the proper management of wastes in a way that reduces pollution of the environment, thus least harm to human health (Allen, 2014). Securing clinical wastes to a bare minimum and reducing the carbon footprints is the key to sustainable dentistry. Recycling reduces the depletion of natural resources and reduces carbon emissions (Thopegowda *et al.,* 2013). It is also true with metallic dental fillings that undergo extraction, purification, and refining for each production batch. Recycling metals and alloys is cost-effective and hugely

*Corresponding author's e-mail: rijesh.m@vit.ac.in

profitable, making them not even a cause in many kinds of research underlining recycling (Baran & Table, n.d.; Kim *et al.,* 2010).

The gold and its alloys regarded as an ideal restorative material have advantages such as resistance to corrosion, good castability, coefficient of thermal expansion compatible with porcelain, and biocompatibility (Knosp *et al.,* 2003). However, the economic burden of using expensive and high-precision noble metal alloys for patients became a major limitation for their application which posed an opportunity for base metal alloys to substitute and deliver the purpose. Over the years, the dental prosthesis has successfully transitioned from gold, platinum, and other precious metal casting alloys to base metal alloys (David, 1982; Huget *et al.,* 1977). Two extensively used base metals in dental prostheses are Ni-Cr and Co-Cr-based alloys. The Ni-Cr alloy possesses good mechanical properties such as high hardness, tensile strength, and low density. Also, the corrosion resistance and good porcelain-metal interfacial bonding of Ni-Cr alloy make the alloy a practitioner alloy for a prosthesis (Tomar *et al.,* 2015). However, the recasting of these metals was not commonly undertaken due to the lack of research and complying with the instructions of the manufacturer suggesting single usage. Studies on the recasting of dental alloys have shown diversified opinions demonstrating complete avoidance of recasting to recasting ten times, causing no change in the alloy's quality (Nelson *et. al.,* 1986; Palaskar *et. al.,* 2010; Sharma *et al.,* 2016). Hence the compositional changes due to the multiple casting became a topic of interest. Reisbick and Brantley (1995) reported no significant diminishment in the tensile properties after recasting type III Gold (46%) alloy up to three times. However, Gupta and Mehta (2012) noticed compositional and physical changes along with decreased fluidity after remelting of base alloys. Harcourt and Cotterill (1965) suggested that the elements such as Co, Ni, Cu, Cr, Sn, Fe, and Zn could alter during recasting due to evaporation and oxidation. Palaskar *et al.* (2010) found no meaningful effects on castability while recasting Ni-Cr alloys.

The studies carried out by Imirzalioglu *et al.* (2012) indicated that alloys containing nickel had increased cytotoxic effects after recasting due to compositional changes. A recast Ni-Cr-Mo alloy in artificial saliva releases more Nickel and Chromium than an alloy that has not been recast (Oyar *et al.,* 2014). Wataha and Lockwood (1998) observed release of elements continues in vitro into the cell culture medium for up to ten months for dental casting alloys. Espevik (1978) found very little corrosion as the Cr content exceeded 16%. The corrosive attack was at Cr depleted grain boundaries. The Cr depletion probably resulted from coring during solidification. Since corrosion and biocompatibility are out of scope in the present study, it only suggests that elemental depletion during recasting is a prime reason for corrosion. Papazoglou *et al.* (1998) suggested that for safe routine clinical implementation of the recast dental alloy many researches need to be carried out to study variation of chemical composition, microstructural and mechanical properties and also porcelain shade. A systematic review carried out by Vaillant-Corroy *et al.* (2015) on the influence of recasting on the quality of dental alloy. The study concluded that there is no consensus protocol to evaluate recasting. Future studies are required for the establishing a standard protocol.

Few manufacturers' product information on Ni-Cr casting alloys typically states that remelting of scrap metals can be acceptable to fabricate clinical castings with 50% of new alloys (Yavuz *et al.,* 2012). The basis for this guideline is the depletion of alloying elements during meting by oxidation. Corroy *et al.* (2015) revealed recasting of base metal alloys could be feasible up to four times, provided remelting in an inert atmosphere. In recent times precision casting technologies where alloys can be remelted without altering composition and other properties by adding the lost elements exist. Hence, the present study aims to evaluate compositional changes of Ni-Cr base alloy (Ni-Cr-Mo) after recasting using a Tungsten Inert Gas (TIG) torch. A tungsten inert gas welding torch melts faster compared to conventional melting in the dental clinic. It represents a bulk melting process. The elemental variation, morphological changes, and mechanical properties of the remelted specimen are studied.

II. MATERIALS AND METHODS

A Ni-Cr-Mo alloy (Ni-Cr-Mo) purchased as pellets were 1.5 cm in length and 0.5 cm in diameter. The elemental compositions of as-received specimens were confirmed using a handheld ED-XRF Spectrometer. As shown in Figure 1, a mould was designed for melting and remelting Ni-Cr-Mo alloy pellets. A cavity of cuboidal shape was within the crucible. The volume of the cavity was slightly more than the volume of the two pellets.

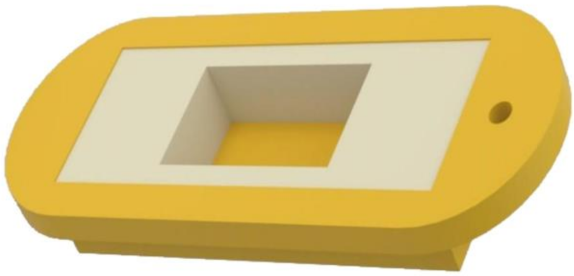

Figure 1. Design of the mould

The mould cavity of cuboidal shape helps achieve a flat surface which is a requirement for ED-XRF spectrometer, metallography, and microhardness measurements. 200 gm of phosphate powder mixed with 100 ml of water is poured inside a crucible boat to prepare the mould. A pattern of styrene was used to form a cavity of 0.7 cm^3 at the centre of the mould. The mould was cured using Nabertherm L24/11 muffle furnace at 900 ^{0}C held for four hours, followed by furnace cooling (Figure 2(a) and (b)). The burnt ash of styrene was blown off. The mould with phosphate powder is a standard practice in dentistry for restricting contamination during pouring and solidification. The Ni-Cr-Mo alloy pellets were melted in the mould cavity.

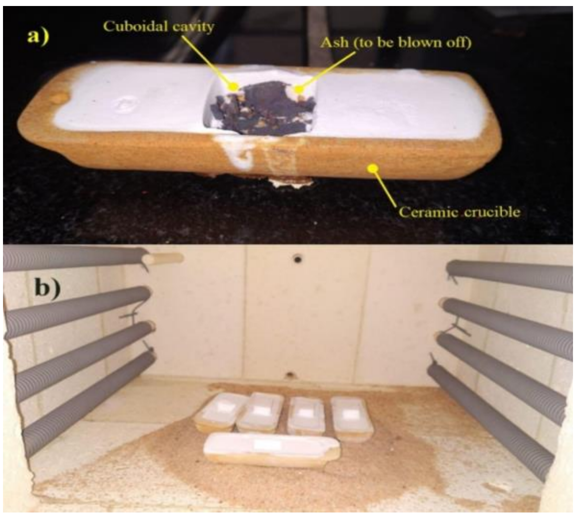

Figure 2. (a) Mould after curing, and (b) Furnace chamber with moulds placed inside for curing

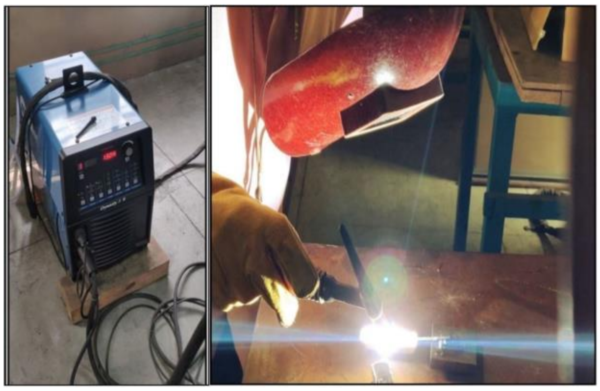

Figure 3. TIG Welding setup and melting of Ni-Cr-Mo pellets using TIG torch

As depicted in Figure 3, inert gas (TIG) torch of a tungsten thoriated electrode with a diameter of 3.2mm was used for melting the pellets. During melting, the channelling of argon gas through the torch helps to protect the molten pool from excessive oxidation. Figure 4 shows the binary phase diagram of Ni-Cr alloy and the ternary phase diagram of Ni-Cr-Mo alloy at 1250^{0}C. The melting temperature for the alloy pellets used in the current study was around 1550^{0}C, and the current and voltages were set accordingly. The samples were melted up to three times to study the change in composition, microstructure, and hardness of the cast specimens. The compositional changes were analysed after each melt. Upon every melting, the used mould is disposed of, and a new mould is used to avoid contamination. The standard mechanical polishing of recast specimens was carried out and etched with hydrochloric acid, acetic acid, and nitric acid solution in the ratio of 1:1:1 to reveal the microstructure under an optical microscope.

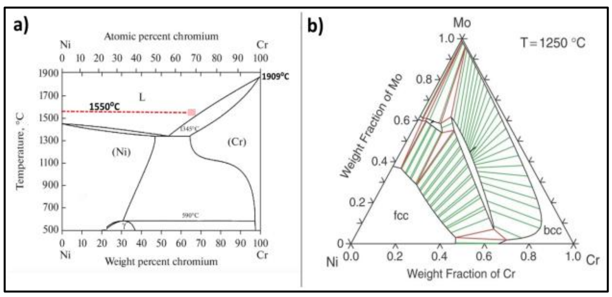

Figure 4. Phase diagrams (a) Binary (Ni-Cr), and (b) Ternary (Ni-Cr-Mo) for selecting the appropriate TIG welding parameters to melt the alloy pellets

The Vickers hardness after each cast was conducted according to ASTM (American Society for Testing and Materials) E-384 on the Mitutoyo HM-200 machine, with a load of 100 gm load and ten seconds dwell time. The average of each sample indented ten different regions was taken as a measure of microhardness.

III. RESULTS

A. Chemical Composition

Figure 5 represents the decrement in the major alloying contents of Ni-Cr-Mo alloy after each recast. It can be seen from Table 1 that the reduction in wt. % of Ni, Cr, and Mo is negligible for the first recast. The depilation in Ni and Mo was 0.46% and 6.1%. However, the Cr was 22.6 wt. % in the base alloy, which remained unchanged.

Table 1. Change in the major elemental compositions after recasting

Elements (wt. %)	As-Bought	First Cast	Second Cast	Third Cast
Ni	65.2	64.9	55	50.2
Cr	22.6	22.6	21.45	20.3
Mo	9.5	8.92	8.13	7.61

The decrease in the concentration of major alloying elements was more severe for the sample, which was recast twice. 15.64%, 5.1%, and 14.42% of Ni, Cr, and Mo reduction compared to the base alloy were found. Furthermore, recasting the alloy thrice has shown a more pronounced loss in the major alloying elements. Depletion of Ni, Cr, and Mo was 23%, 10.18%, and 19.89%, which is 36%, 5.08%, and 5.77% higher than recasting twice. Table 2 represents a theoretical analysis of elemental compositions of alloys used in the present study mixed in various proportions. As the proportion of the as-received alloy increases, the deviation from nominal composition decreases. That is probably why many researchers have suggested mixing with a higher percentage of new alloy (Ayad, 2002; Hesby *et. al.,* 1980; Jochen *et. al.,* 1991; Nandish *et. al.,* 2020; Pseraire *et al.,* 2007). Nevertheless, none of the proportions could recast Ni-Cr-Mo alloy with a composition equivalent to received conditions. A similar trend was observed with the second and third recast too.

The elemental composition depleted after each recast in the present study is calculated and represented in table 3. For instance, the depilation of Ni after the first, second, and third recast was 0.3, 10.2, and 15 wt. %. However, the depilation of Cr and Mo after consecutive recasting was 0, 1.15, and 2.3 wt. %, and 0.58, 1.37, and 1.89 wt. %, respectively.

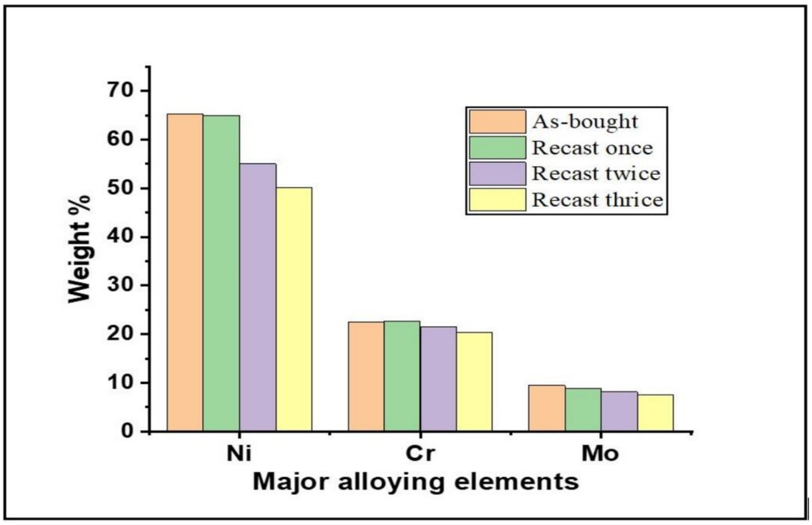

Figure 5. Composition changes in the Ni-Cr-Mo alloy with each of the recasting (wt. %)

Table 2. Theoretical composition analysis by mixing the recast alloy with various proportions of base metal alloy

	Recast Once			Cast Twice			Cast Thrice		
	Ni	**Cr**	**Mo**	**Ni**	**Cr**	**Mo**	**Ni**	**Cr**	**Mo**
25%	64.96	22.6	9.036	57.04	21.68	8.404	53.2	20.76	7.99
50%	65.0	22.6	9.113	58.4	21.83	8.59	55.2	21.07	8.24
75%	65.02	22.6	9.168	59.37	21.94	8.72	56.63	21.29	8.42
85%	65.04	22.6	9.19	59.69	21.98	8.76	57.09	21.36	8.48
100%	65.05	22.6	9.66	60.1	22.03	8.82	57.7	21.45	8.56

Table 3. The wt. % of major alloying elements for the master alloy

	Ni (wt. %)	**Cr (wt. %)**	**Mo (wt. %)**
If cast once	0.3	0	0.58
If cast twice	10.2	1.15	1.37
If cast thrice	15	2.3	1.89

The elements lost after each recast, along with some extra (to compensate for evaporation loss), can be added during the recasting of nominal composition. A simple elemental analysis and calculation of lost elements will help in designing the master alloy. A master alloy is a raw material that's alloyed with various percentages of elements. They are cast into shapes that help to diffuse to the molten alloy at lower temperatures. It is a convenient way of adding small quantities of elements to the alloy melt with significant differences in melting point. Master alloys change the composition of molten alloy to achieve a specific property, such as mechanical strength, biocompatibility or to reach a particular chemical specification. For example in this study, the depletion in Ni, Cr, and Mo was 0.46%, 0%, and 6.1% in the first recast. So a master alloy has to be prepared with only Ni and Mo with a nickel content of 0.3% and Mo content of 0.58% more than the as-bought alloy so that the lost elements are replenished. Similarly, a master alloy can be prepared for any loss in elemental composition and recast. Depending upon the quantity to be added, the % increase in elements can be varied. The amount of each element to be added will vary based on the number of recasting.

B. Microstructure Development

Figure 6 represents the optical microscopic images of the Ni-Cr-Mo alloy recast for once, twice, and thrice, respectively. The micrograph shows a typical cast structure with well-developed dendrites with arm spacing. No drastic variation in the microstructure was observed for the alloy with several melting and solidifying cycles. This statement was in line with a study done by (Nelson *et al.,* 1986) that combining excess used metal with new metal and recasting for up to ten times revealed no significant degenerative changes in physical properties, microstructure, and clinical characteristics.

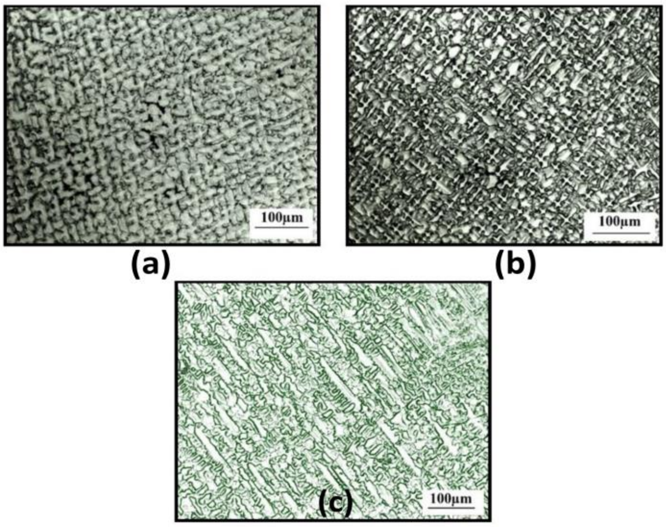

Figure 6. Microstructure of recast Ni-Cr-Mo alloy, recasted (a) Once, (b) Twice, and (c) Thrice

C. Hardness

Figure 7 represents the microhardness measured for the as-received and recast Ni-Cr-Mo alloys. No substantial increment or decrement in hardness took place up to three castings. This outcome confirmed to results obtained by Bauer *et al.* (2010) on evaluating Ti in as-received and recast conditions on the mechanical properties.

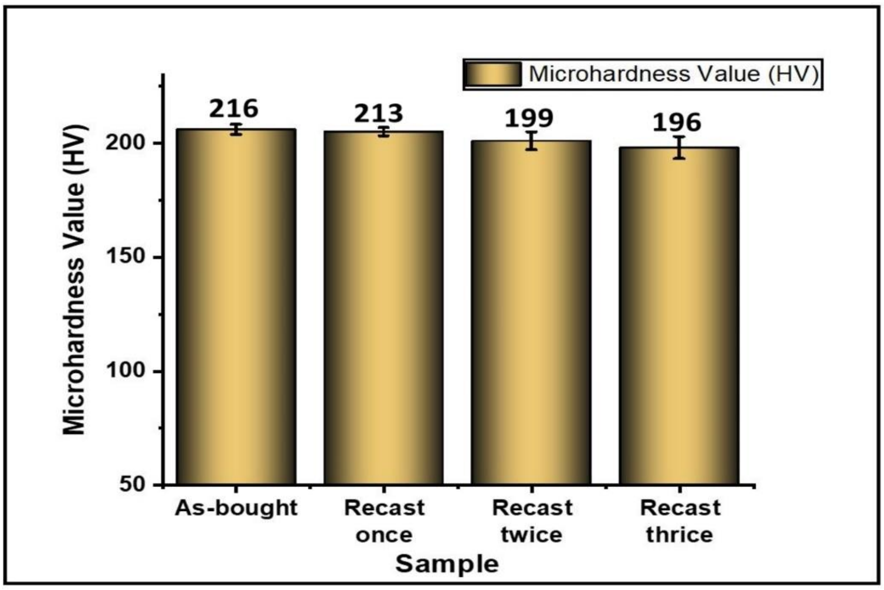

Figure 7. Microhardness values of Ni-Cr-Mo alloy after recasting once, twice, and thrice

When the commercially pure Ti alloy was recast, no determinantal variation in the mechanical properties were noticed, even though the alloy has susceptible to contamination during recast. However, the results obtained by Walczak *et al.* (2019) revealed a decrease in the mechanical strength and an increase in the heterogeneity in the structure of the recycled Co-Cr-Mo alloys resulting from the precipitation of various types of carbides and the presence of intermetallic phases.

IV. DISCUSSION

A. Chemical Composition

The oxidation or vaporisation may be the reason for the elemental loss; the oxides form part of the slag. The melting loss is the difference between the cold metal charge and the finished melt. This is higher when small parts like sprue gate and other casting surplus are melted instead of compact scraps.

The previously published works of literature on the recasting of dental alloys differ widely, with outlooks ranging from restraining recasting to recasting up to ten times without any changes in the alloy's quality. Various researchers recommended 50% new alloys to the previously melted alloy (Ayad, 2002; Hesby *et. al.,* 1980; Imirzalioglu *et. al.,* 2012; Nandish *et al.,* 2020). Bauer *et al.* (2010) and Jochen *et al.* (1991) predicted the loss of secondary elements by evaporation and oxidation. Lin *et al.* (2013), Liu *et al.* (2010), and Lopes *et al.* (2005) found that the adherance of porcelain may decrease with the depliation of elements. Mirkovic *et al.* (2008) recycled alloy residues through twelve casting generations with 50% of new alloy on the occasion of every recasting. They had an unfavourable effect on the thickness of the metal-ceramic interface of the examined alloys.

Yavuz *et al.* (2012) conclude that repeated recasting of Ni-Cr alloys (as-received metal, 50 wt. % new metal, 50 wt. % once-recast metal, and Group 100% once-recast metal) causes changes in the elemental composition, particularly Cr due to aluminium oxide sandblasting. Rasmussen and Doukoudakis (1984) investigated the effect of recast gold alloy to study metal-ceramic fracture surfaces. The authors reported that there were no adverse effect on 75% recast Olympia, eventhough a small change took place for 85% or more recast alloy. Jochen *et al.* (1991) investigated the effect of reusing a silver-palladium alloy in 0%, 25%, 50%, 75%, and 100% combinations of new and once-cast alloy. The four-point bending test was conducted, and the results showed that at least 50% new metal is needed every time to retain its original flexural strength. However, attaining composition equivalent to as received with additions of received alloys in any proportions is not feasible.

So one can infer that mixing in various proportions is purely empirical and will not yield the same composition as the parent metal. For obtaining the same composition, a chemical analysis of the recast sample is necessary. The elements added as master alloy prepared concerning the elements depleted in the exact proportion gives exact proportion.

Madani *et al.* (2011) stated that although it is a common practice to recast base metal alloy, recasting of Ni-Cr and Co-Cr alloys have an unfavourable effect on metal-ceramic bond strength. The increase in the thickness of oxide layer is the main factor which diminishes metal-ceramic bond strength in the base metal alloy. However, Yilmaz *et al.* (2012) concluded that clinicians could use 100% recast metal without any adverse effect on opaque porcelain colour. Ucar *et al.* (2009) suggested that recast base alloy with new alloy decreases metal-ceramic bond strengths due to oxidation. From the above studies, it is clear that the decrease in bond strength with recast base metal alloy is not due to the decreased elemental composition but to increased oxidation which is controllable in an inert atmosphere.

B. Microstructure Development

Microstructural developments after solidification are resultant of nucleation and growth. During growth, the redistribution of solute at the solid and liquid interface determines the stability of the solidification. The solidification begins with the planar solid/liquid interface. The thermal and composition gradients at the interface govern the morphological stability of the planar solid-liquid interface. In Ni-Cr-Mo alloys, nucleating sites were present. The temperature distribution in the liquid melt creates a temperature gradient that generally arises from the heat flow condition is one of the critical factors for the microstructural evolution. A smaller thermal gradient supercools the liquid ahead of the interface, and the actual temperature becomes lower than the equilibrium liquidus temperature. Perturbation of the plane front will not melt; instead, that will grow as cellular or dendrites. A similar dendritic structure is seen in the present study too.

C. Hardness

The dependence of the microhardness on dendritic growth velocity is directly related to the grain size variation caused by the rapid dendrite growth from the undercooled melt. Dendrite coarsening can occur while dendrite refining with the increase of undercooling. In our experiment, dendrites continuously refine with undercooling, and no dendrite coarsening phenomenon is found. Since the average grain size and dendritic growth velocity is almost constant for all recast, a difference in hardness was not observed in this study (Figure 7). However, few researchers like Peraire *et al.* (2007) noticed a significant increase in Vickers hardness appearing in the fifth recast. Gupta and Mehta (2012) reported that repeated remelting of base metal alloy for dental casting without adding new alloy will affect the alloy properties. The increase in hardness reported in the work maybe due to intermetallic and carbide formation. The dental alloys used in restorations should not contain carbon. The carbon that comes from the atmosphere needs restrictions during remelting. The intermetallic formation in ternary alloys can also be engineered by proper heat treatment. Therefore, if appropriate care is taken, a drastic change in mechanical property is not possible even after the elemental loss during remelting.

V. CONCLUSION

Based on the analysis of results obtained from chemical analysis, microstructure, and hardness of Ni-Cr-Mo alloy before and after recasting, the following conclusions can be drawn:

Added quantity of previously cast alloy to a new alloy to compensate for lost elements is empirical and will not yield the same composition as the new alloy. Attaining the exact concentration is possible only by a chemical analysis of the recast sample and adding elements in the form of a master alloy prepared according to the elements depleted in the exact proportion.

The number of recasting can be as many times provided depleted elements added in exact proportion after each recast.

The microstructures show a typical cast structure with well-developed dendrites with arm spacing. No drastic variation in the microstructure was observed for the alloy with several melting and solidifying cycles.

Substantial increment or decrement in hardness was not observed up to three castings due to melting in a controlled atmosphere and not facilitating carbides and intermetallic formation.

The broad implication of the present research suggests recycling dental alloys in bulk outside a dental clinic. A dentist should responsibly segregate different base metal alloys and promote sustainable dentistry.

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