A Comparative Analysis of the Influence of New and Recast Alloy Combinations on Castability, Chemical Analysis, Microstructural Properties of Ni-Cr Alloy

G.Gomathi*, Y.A.Bindhoo**, C.Sabarigirinathan***, E.Subramaniam****

*Associate Professor, Department of Dental Surgery, Stanley Medical College & Hospital, Chennai-01 **Past postgraduate student, TamilnaduGovt Dental College & Hospital, Chennai -03 ***HOD,Dept of Prosthodontics, TamilnaduGovt Dental College & Hospital, Chennai -03 ****FormerHOD, TamilnaduGovt Dental College & Hospital, Chennai -03 Corresponding Author – Dr G.Gomathi MDS,

Abstract:

Statement of The Problem: The reuse of casting alloys has become customary for the economic reasons; the proportion of the fresh and reused alloysis a concern to maintain the properties of the casting restoration.

Materials & Methods: Ni-Cr dental casting alloys are taken up for the study in a various combinations of fresh and recast alloys, and tested for castability, changes in chemical composition and microstructure. The alloy combination was grouped in four divisions, 100% fresh alloy, 50% fresh alloy with 50% recast ally,25% fresh alloy with 75% recast alloy and 100% recast alloy. A total 24 samples were prepared for castability with six samples in each group, and a single sample for each group to test the chemical analysis and microstructure evaluation. Hinmann's counting method for castability, optical emission spectroscopy for chemical composition analysis; light and scanning electron microscope (SEM) for microstructure evaluation were used in this study.

Results: The use of high percentage of recast alloys Group III & Group IV have a definite effect on the castability but within the limit, a gradual change in chemical composition of certain elements like C,Cr,Si,Mn, and the presence of continuous carbide precipitation in microstructure evaluation.

Conclusion: Within the limitations of the present invitro study, the use of lower percentage combinations though produced statistically significant difference in the alloy's properties, but was within the required minimum limits.

Keywords: castability, chemical analysis, microstructure evaluation, ni-cr alloy, recast alloys.

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I. Introduction

Several factors relating to both the material &technique employed in investing and casting procedure influence the properties of cast restoration. One such factor is the use of recast alloys for economic reasons. Casting procedures usually require the use of more metal than is needed to produce a restoration. In the dental laboratory, the surplus alloy is commonly reused along with new alloy. The judicial combination of new and recast alloy should be cost effective but at the same time should not compromise the properties of the resultant casting. Thus a thorough knowledge and understanding about the influence of recast alloys on castability, the changes in chemical composition and microstructure are essential to have a predictable outcome.

Dental alloys can undergo degradation upon recasting due to their complex chemical composition.^[1, 2] They contain elements, which can oxidize, absorb gases or adhere to crucible walls during melting & casting procedures. As a result, the concentration of these elements can decrease with every casting. Consequences of the above reactions can have an impact on the microstructure of the alloy like porosities, alteration in phase structure, and grain size. These microstructural defects may decrease the corrosion resistance and result in fatigue failure of the cast restoration when used in oral cavity.^[1,3,4] A great concern exists with remelting alloys for metal ceramic restoration, because of the potential loss of trace elements having important role in metal ceramic bonding, tensile strength, castability, corrosion resistance and other properties. Hence the profitable use of recast alloys can be done, by using, properly cleaned recast alloy, by properly balancing old & new alloy and by reducing alloy damage by avoiding excessive remelting at high temperature or using carburizing flame. Mc Lean^[5]& others ^[6, 7]have suggested that at least 50% new metal be included when using recast alloys. The aims of this in-vitro study were to analyze the influence of new & recast alloys combinationson the following properties.

Castability Chemical composition Microstructure evaluation

II. Materials & Methods:

The alloy evaluated in the study was 4-all Metal white ceramic alloy (IvoclarVivadent) with the following composition Ni-61.4%, Cr-25.7% Mo-11.0%, Si=1.5%, C=<1.0%. The total samples were grouped in four categories based on the percentage of fresh and recast alloy used

Group I: Cast with 100% New AlloyGroup II: Cast with 50% New and 50% Recast Alloy

Group III: Cast with 25% New and 75% Recast AlloyGroup IV: Cast with 100% Recast Alloy

2.1 Preparation of Samples.

The samples were prepared as per the requirement of the tests evaluated. Twenty four samples for the castability, four samples for chemical analysis and four samples for microstructure evaluation were prepared. For castability six samples per group and for chemical and microstructure evaluation one sample per group was prepared. The castability monitor employed in the present study follows the standard method of castability proposed by Whitlock & Hinman (1985), in which the potential of dental alloys to reproduce a mesh of nylon net was verified & used as a castability standard. The dimensions of the mesh monitor are in accordance with ASTM E 11- 70 specification as used in earlier studies. The mesh patterns in this study were fabricated from polymer (Netlon,India). The dimensions of the mesh pattern were 10x10 filaments comprising 220 segments with 100 open squares of approximately 1mm opening and a nominal filament diameter of 0.35mm. Four circular discs of approximately 12mm diameter and 3mm thickness were prepared using autopolymerising acrylic resin for each group of chemical analysis.Four cuboidal resin blocks of approximately 10mm length and 10mm height were prepared for each group of microstructure evaluation.

2.2Spruing, Investing and Casting of Samples

All the patterns were sprued and invested using phosphate bonded investment(Flex vest - Ivoclarvivadent) in a wet ring liner lined casting ring. All of the recast alloys used in the present study were once used alloys, and sandblasted for cleaning it to avoid variation. The new & recast alloys required for each group was weighed using an electronic weighing machine (Fc series) as required for each group. The amount of alloy (New or recast) required for each casting was determined by the following formula.

Gram of alloy required = weight of pattern x density of alloy (8.4g)As specified by the manufacturer

Separate crucibles were used for each group to avoid alloy contamination. The whole casting procedure was carried out in as short a time as possible using spring loaded casting machine and gas-oxygen torch. The casting rings were allowed to cool to room temperature & then divested. The castings were then retrieved and sandblasted using 50μ grit aluminium oxide.

2.3Finishing of cast samples

3.1 Castability

For evaluation of castability and chemical analysis, the specimens were sandblasted. For microstructure analysis the samples were embedded in Phenolic mounting compound (Bakelite) to allow easy finishing and polishing of the alloy surface. The specimens werepolished at intervals until a smooth reflecting scratch free surface was obtained. The samples were chemically etched using the etchant, Marbles' Reagent (10gm CuSO4 (copper sulfate), 50ml HCl, 50 ml H₂O) by immersing the samples for 30 seconds to produce a correct observation of morphologies.

III. Evaluation & Testing

The procedure for assigning a numerical value for castability of the cast specimen was done according to the methodology described by **Hinman et al**. The square shaped mesh pattern provides a grid with 100 open squares & 220 segments. The numbers of completely reproduced cast segments were counted. The castability is designated as a percentage referred to as castability value. It is calculated as follows;

Number of completely reproduced cast segments

X 100

Castability (%) = ____

220 (total number of segments)

This indicates the accuracy of the alloy to reproduce the wax pattern details. The criterion for determining complete & incomplete segments is illustrated in [Fig 1-]. Segments were considered incomplete if they did not completely extend from the far edge of a crossing segment to the far edge of the next one. The cast meshwork samplesto be counted in [Fig 2]

3.2 Chemical Analysis

The circular alloy samples cast according to the specified alloy combination of each group were subjected to chemical analysis using optical emission spectrometry (Model – Metallab, GNR, Italy). The optical emission spectrometer offers rapid elemental analysis of solid metal samples. This method involves statistical processing of the spark pulse-generated emission spectra obtained from spark discharges in an argon atmosphere. The first part is an electrical source to excite atoms within a metallic sample so that they emit the distinctive light (element- characteristic emission lines). The second part is an optical system with diffraction grating spectrometer that separates the incoming light into element-specific wavelengths and a corresponding detector measures the intensity of light for each wavelength. The third part is a computer system which processes the measured intensities via predefined calibration to produce elemental concentration, the results are displayed in the monitor which can be printed or saved.

3.3 Microstructure Evaluation

The embedded cuboidal alloy blocks were subjected to metallographic examination using light optical microscope (Metscope – I, under 100x and 400x magnification) and Scanning Electron Microscope (JSM-840A SEM, JEOL-JAPAN, less than 1000x and 4000x magnification).

IV. Results

4.1 Results of Castability

The castability was calculated by counting the completely reproduced segments and given in Table-1. The values are higher in 91.48% in Group I followed by Group II, III, and the least was in Group IV 72.55%.

4.2Results of chemical analysis

The results of the chemical analysis indicates that although not very significant changes occurred in the alloy composition, there is a gradual reduction in the percentage of certain elements when the percentage of recast alloys was increased. Chromium, Carbon, silicon and manganese content decreased from group I to group IV but that of the Molybdenum is increased.

4.3Results of microstructure analysis

The microstructure of Ni-Cr alloy evaluated in this study revealed a dendritic morphology accompanied by compositional precipitation of carbides. The microstructure of group I under light microscopic view showed fine dendritic pattern of matrix phase with carbide islands. The SEM view revealed very few micro porosities [Fig-3]. The group II was similar to group I except for the dendritic structure being slightly coarser with more carbide precipitates [Fig-4]. The group III showed large dendrites with carbide precipitation along the grain boundaries under light microscopic view. The SEM view shows lamellar arrangement of carbidesalong grainboundaries with more micro porosities and inclusions than group I & II[Fig-5]. The microstructure of group IV showed extensive networking and lamellar arrangement of carbides along the grain boundaries and a coarser dendritic structure. The SEM view reveals the extensive networking of carbides and increased micro porosity [Fig-6].

V. Statistical Analysis

The values were statistically analyzed using,One-way ANOVA, Duncan's Multiple range test.In the present study, the within group variance is found inside group I, II, III, IV, individually. Between groups variance is found between 4 groups. Thus the groups were compared within themselves & also against each other. As the P value for all the properties evaluated was less than 0.001 there is a significant difference between the groups with regard to various percentage of alloy combination. There was a significant difference in castability among the 4 Groups tested. Castability of group I (91.48%) was significantly higher than other groups (Group II (88.8%), group III (83.75%), & group IV (72.55%). The mean value of the all the groups for each property was then compared by using Duncan multiple range test calculated at 0.05% significant at 5% level.

VI. Discussion

One of the problems associated with any method evaluating castability is that, small variations in casting technique may have significant effects on the results obtained. Whitlock & Hinman¹ have proposed that evaluation of castability should be accomplished by comparing results obtained from castings made under identical circumstances rather than by comparing data obtained elsewhere under different circumstances. The findings of the in-vitro study indicated that, under the tested experimental conditions, the castability of group IV (72.05%) was significantly less than the other groups suggesting that the use of 100% recast alloys will influence the castability of these alloys. It was noticeable that the alloy became less fluid as the recast alloy

percentage increased. Similar results were reported by Mosleh, Abdul-Gabbar⁸who assessed castability using new & recast alloys in the ratio of 50:50. The results revealed superior percentage castability for gold alloys, with comparable results produced by Ni-Cr alloy.

The results of the chemical analysis indicate that although not very significant changes occurred in the alloy composition, there is a gradual reduction in the percentage of certain elements when the percentage of recast alloys was increased. Chromium, Carbon, silicon and manganese content decreased from group I to group IV but that of the Molybdenum increased.Similar results were reported in earlier studies,^{9, 10,11,12,13} the changes are minimal they can become more pronounced with repeated remelting, altering the composition of these alloys. Eugene F Huget, Jesus M. Vlica¹⁰ (1978) in their study on the ceramometal alloys explain that modifications of the Ni-Cr alloy by minor alloying element have diverse structural features and properties

The microstructure evaluation of the present study shows that the carbides were seen as discontinuous precipitates in group I (100% new alloy) & group II (50% new & 50% recast alloy) but found to form a continuous network along the grain boundaries in group III (25% new & 75% recast alloy)& group IV (100% recast alloy)in light microscopic view. The microstructure revealed a gradual loss of refinement of dendritic structure as the percentage of recast alloy was increased. The SEM view of the groups evaluated showed a gradual increase in microporosity suggesting that casting defects increases as recast alloy percentage increases.Carbide precipitates dispersed between the matrix phases was a common occurrence in all the groups. Asgar¹⁴& Harcourt ¹²reported that spherical, discontinuous carbides seen in Co-Cr alloy resulted in increased percentage elongation and tensile strength. Carter suggested that large continuous carbides produce a path for crack propagation retarding the mechanical properties of Co-Cr alloy. ¹⁵The carbide structure is known to have a definite effect on the physical properties of the alloy.¹²Harcourt^{12,13} stated that, excessive remelting of the alloy leads to some changes in the composition, physical properties & microstructure, these changes becoming significant following a larger number of remelts.It can be inferred from this study that the proportion of fresh and reused alloys have a definite effect on the bonding ability and chemical composition and microstructural level as the last two factors have an effect on the bonding ability and physical properties.

VII. Conclusion

Within the limitations of the present invitro study, the following observations were made;

- 1. Castability was affected when 100% recast alloy was used (group IV). Minimal changes were seen with 75% recast alloy (group III) & 50% recast alloy (group II).
- 2. The chemical analysis of the groups revealed a gradual change in the alloy constituents namely, Chromium, Carbon, Molybdenum, and Silicon as the percentage of recast alloy steadily increased from group II (50% new alloy&50 % recast alloy) to group IV (100% recast alloy).
- Microstructural analysis revealed a gradual loss of refinement of dendritic structure & increased microporosity and carbide networking as the percentage of recast alloy steadily increased from group II (50% new alloyand50 % recast alloy) to group IV (100% recast alloy).

From the results of this invitro study a Porcelain Fused Metal coping of up to 50% recast alloy appears to be an adequate safety margin. But use of recast alloys up to 75% can be considered provided alloy contamination by excessive melting or inadequate cleaning are reduced and proper investing and casting steps are carried out to minimize casting defects. Further investigation regarding the corrosion resistance, cytotoxicity of this alloy should be evaluated for better.

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Chart 1 – Bar Diagram of Castability of Four Groups

Table -1 castability value **- number of segments reproduced, *** castability value in percentag	Table -1 castabilit	v value **- number	of segments reproduced.	. *** castability value	in percentage
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C N-	GROUP I		GROUP II		GROUP III		GROUP IV	
S. No	**	***	**	***	**	***	**	***
1.	203	92.2	195	88.6	189	85.9	151	68.0
2.	200	90.9	196	89.0	186	84.5	162	73.6
3.	202	91.8	197	89.5	183	83.1	169	76.8
4.	198	90.0	195	88.6	183	83.1	155	70.4
5.	201	91.3	196	89.0	178	80.9	157	71.3
6.	204	92.7	194	88.1	187	85.0	159	72.2
Mean		91.48		88.80		83.75		72.55

Table -2 One WayAnova & Duncan Multiple Range test - in superscript

S.NO	GROUPS	MEAN	SD	P VALUE
1	Ι	91.48 ^d	1.15	< 0.001**
2	II	88.80 ^c	0.48	
3	III	83.75 ^b	1.77	
4	IV	72.55 ^a	2.59	

Different alphabets denote that values are significant at 5% level

Table 3.- Results of chemical analysis of various Percentages of new and recast alloy combinations

S.NO	Composition of the alloy	Composition (%)				
	pellet	Group I	Group II	Group III	Group IV	
1.	Chromium	20.308	20.36	19.75	18.48	
2.	Silicon	1.328	1.288	1.264	1.239	
3.	Molybdenum	11.6	11.21	12.77	12.05	
4.	Carbon	0.07	0.06	0.044	0.034	
5.	Aluminium	0.015	0.016	0.016	0.015	
6.	Manganese	0.083	0.085	0.28	0.33	
7.	Magnesium	0.029	0.030	0.030	0.029	
8.	Niobium	0.07	0.075	0.34	0.30	
9.	Iron	1.231	1.024	0.70	0.65	
10.	Nickel	Balance	Balance	Balance	Balance	

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MICROSTRUCTURAL ANALYSIS A-100x LM , B-400xLM , C-1000x-SEM, x- Magnification,LM-Light Microscope, SEM- Scanning electron microscopy



Figure 3 GROUP I – carbide precipitation in a well-defined matrix



Figure 4 GROUP II – carbide precipitation along the grain boundaries



Figure 5 GROUP III – carbide networking along the grain boundaries & inclusions



Figure 6 GROUP IV -continuouscarbide networking along the grain boundaries and inclusions

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