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Effect of addition of recast materials on characteristics of Ni-Cr-Mo alloys

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ABSTRACT

The purpose of this study was to assess the effect on selected mechanical properties, of adding recast materials to the NiCrMo alloy of newly produced castings.

Three groups of dental alloy NiCrMo (trade named Remanium CS+) were prepared by mixing 50% new alloy to alloy remnants from previous castings. The specimens in the first casting group used 100% new alloy and served as control (C1). The second group consisted of equal amounts of new alloy and alloy remnants cast only once (C2). The third group contained 50% of new alloy and alloy cast twice (C3). Microstructural analysis was performed and the chemical composition, hardness and the metal-ceramic bond strength were assessed. In addition, EDS analysis (mapping) was undertaken. Hardness and bond strength results were also statistically analysed.

In spite of the fact that recasting brought about small changes in hardness and chemical composition (C, Cr and Mo), these effects were found to not affect their functional properties in the oral cavity. Still, significant differences between new alloy and the recasted groups ($p < 0.05$) were demonstrated in the course of statistical analysis of Vickers hardness test (for $\alpha = 0.05$). All analysed research groups have a similar average adhesion at 48.51÷49.24 MPa ($p > 0.05$).

The recasting procedure described in the paper can be done safely in dentistry. If previously casted material is used, it should be mixed with new material. The use of the material prepared in this way can lower the costs of NiCrMo castings.

INTRODUCTION

Nickel alloys are commonly used in dentistry as base for crowns made with metal faced ceramics and represent a good alternative to expensive precious alloys [1,2]. In addition to economic reasons, nickel-based alloys have the advantage of increased modulus of elasticity in comparison with gold. This allows the use of thinner cross sections and consequently lower damage to the healthy tooth during crown constructing [2]. Moreover, the coefficient of thermal expansion of nickel-based alloys is compatible with the thermal expansion coefficient of conventional veneer ceramics, thus a close connection between the metal crown and ceramics is provided during the firing process, hence the possibility of the veneer cracking is lessened [3]. Because of the above properties, nickel-based alloys have grown in popularity and demand for such base metal products has increased the prices of these raw materials [4]. In order to reduce the cost of prosthetic apparatuses, the process of re-melting earlier cast alloy has become a routine procedure in laboratories.

Residues from the casting process of skeletons (e.g. from the channels of casting mould or from casting cones) and badly made castings (e.g. with short run castings, shrinkage porosities and cracks) constitute prosthetic "scrap". In prosthetics, this scrap is frequently used as a part of charge for recasting [5]. In the mouth during the chewing process, varying degrees of stress concentration occurs in the hard tissues of the tooth and prosthetic restorations. This may result in damage to the fastening elements of the prosthesis or shattering of the ceramic phase of prostheses permanently veneered with dental porcelain [6,7].

In practice, a huge number of producers of casting dental alloys allow the use of 50% of added material from recasts to the new factory alloy. However, a group of manufacturers

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reserve the fact that the addition of so-called scrap can be recast only once and all must come from the same batch. Furthermore, other manufacturers do not allow for the use of additions in the form of repeatedly recast materials or do not provide any information on possibilities of use of post-production scrap.

In dental prosthetics, Ni-Cr [4] and Co-Cr [7] alloys [1], as well as gold-based alloys [8] re-use seems to be promising. However, literature data [4,9,10] indicate that many properties of the recast alloy may differ from the new alloy that was purchased from the manufacturer. These differences may involve chemical composition of alloy [10], castability [4] and mechanical properties [5]. As regards changes of mechanical properties, the opinions are particularly strongly divided. These changes largely concern alloy hardness and tensile strength. According to some authors [9,11], these properties may increase or decrease [5,12]. However, researches conducted by Palaskar *et al*. [4] demonstrate that recasts do no engender statistically significant change in the castabilities of recast alloys.

In the literature, there are experimental studies [6,7,10] that demonstrate a change in chemical composition of final products under the influence of successive recasting processes and the development of new phases determining the changes in alloy properties. Herein, the change in chemical composition may impair the corrosive properties of recasts [13], and may have an impact on cytotoxicity [14].

In addition, the process of applying recast materials may result in a change in the composition of metal oxide surface, which may be critical for bonding between metal and ceramic material [15].

An important aspect of sustainability of prosthetic metal-ceramic apparatuses is sufficient adhesion of porcelain to metallic structure [3,6,7,16]. Ucar *et al*. [16] do not recommend use of recast alloys, as this can reduce the strength of bond between metal and porcelain.

Therefore, the aim of this study, i.a., was to assess the effect of addition of recast materials on the strength of metal-ceramics bond of newly produced castings of NiCrMo alloy. This condition might causes different degrees of stress concentration in hard tooth tissues and prosthetic restorations which may result in damage of fastening elements of prosthesis or spalling of ceramic phase from prostheses permanently faced by dental porcelain.

MATERIAL AND METHODS

Remanium CS+ dental alloy (Dentaurum, Germany) with nickel matrix and nominal Remanium CS+ (w/w) consisting of 61% Ni, 26% Cr, 11% Mo, 1.6% Si, Co < 1%, Fe < 1% and Ce < 1% were used in the tests. This alloy is employed for casting denture elements (among others, crowns and bridges) with permanent ceramic veneers. Three groups of identical dental alloy were prepared in order to simulate standard applications used in dental laboratories. The first group was cast in 100% new alloy as a control group (C1). The other groups were made of 50% new alloy and 50% remnants from the previous group (Table 1). Here, the second group (C2) used a mixture of equal amounts of new alloy and alloy leftover from previous recasting. The alloy used in the third group (C3) was prepared using new alloy and 50% remnants from the second group (re-re-castings) (C2).

Table 1. Different recast alloy groups used in this study

Groups	Procedure
Never recast (R1)	Cast 100% new alloy
Cast once (R2)	Cast from 50% new alloy and 50% remnants from first group
Cast twice (R3)	Cast from 50% new alloy and 50% remnants from second group

The process associated with preparation of specimens made of NiCrMo alloy was carried out in conditions existing in professional prosthetic dental laboratories in accordance with procedures utilized for production of metal denture elements. The castings were produced by applying the investment casting process by means of a vacuum-pressure casting machine Nautilius (Bego, Germany) and ceramic crucibles.

The specimens used for testing hardness and chemical composition analysis were made as discs with diameter of Ø 25 mm and thickness of 2 mm. The discs were then subjected to grinding by means of water abrasive papers with grain size of 220, 600 and 1200 correspondingly. After grinding, the specimens were mechanically polished by means of diamond particle suspension 3 μm and oxide particle suspension 0.05 μm, and subsequently washed in acetone and dried thereafter.

The hardness of tested materials was measured under the load of 98.07 N on a FV-700 Vickers hardness meter with automatic ARS 900 system manufactured by Future-Tech Corp. Forty (40) hardness measurements were performed for each group of specimens.

The analysis of chemical composition was performed by means of a Q4 Tasman 130 spark emission spectrometer (Bruker, Germany) and applying the Ni110 testing channel. Herein, five (5) analyses (sparking sequences) were undertaken for each specimen.

The specimens for ceramic bond strength tests were prepared in accordance with requirements listed under the PN-EN ISO 9693 [17] standard in the form of rectangular plates with dimensions of 25×3×0.5 mm. IPS d.SIGN dental porcelain (Ivoclar Vivadent, Schaan, Liechtenstein) with dimensions of 8×3×1 mm was applied centrally onto the metal plates. The whole process associated with dental porcelain fusion to metal was carried out in conditions existing in professional prosthetic dental laboratories. Nine (9) specimens from each test group were used for testing. The three point-bending test was carried out by means of a Zwick Z100 universal testing machine equipped with 500 N measuring head. The distance between the supports was equal to 20 mm and the diameter of specimen supporting rollers was equal to 2 mm. The testing speed (traverse beam feed) was equal to 1.5 mm/min. The loss of bond strength was indicated by the value of force at which a load disturbance (reduction) was observed in a deflection curve. The bond strength (τ_b) was then determined using the following formula [17]:

$$\tau_b = k \bullet F_{fail} \quad (1)$$

where: k – the factor depending on thickness of base metal and Young module, F_{fail} – the force causing the loss of metal-ceramic bond strength.

The microstructure of tested materials tests was analysed by means of a Phenom G2 Pro desktop scanning microscope.

TESTS RESULTS AND DISCUSSION

Figure 1 shows the microstructure of Remanium CS+ alloy cast by vacuum-pressure. This is characterised by having a typical dendritic structure, but against the background of a solid solution rich in Ni, interdendritic areas are visible. These are observed as unbundling eutectic phases rich in Mo and characterised by their irregular shape (Fig. 2). In the immediate vicinity of the interdendritic phases, an increase in Cr is notable. The area of the dendrites is also dominated by Ni and Cr, and depleted in Mo. There was no substantial differences in shape of interdendritic phases between recast alloys in 100% new material and alloys with the addition of recasts, although XRD analysis as presented in [6], showed differences in the phase composition. Herein, intermetallic phases Mo0,24 Ni0,76 and CrNi2 coexisted, and also Cr7Ni3 of a different percentage share was noted depending on the type of casting.

Wylie *et al*. [2] saw similar results in the EDS analysis of structures of Ni-Cr-Mo alloy (Matchmate NP). They claimed that in the vicinity of the particles in the interdendritic areas, Cr was slightly enriched, while Mo was reduced. Moreover, in the area of the dendrites, Ni enrichment occurred, Mo content decreased slightly and Cr content did not change. Changes of chemical composition of tested materials are shown in Table 2. The manufacturer – Dentaurum company does not demonstrate in their data any information on elements concentrations of less than 1%. We saw a slight increase in carbon content for C3 alloys with 50% addition of cast twice material, as compared to other research groups. This effect can further determine the increase in hardness for this group of materials. There was a also a slightly lower average level of molybdenum ~ 0.3÷0.51% and chromium 1.55÷2.51%, compared to what is declared by the manufacturer.

Chromium is the main alloying element in alloys having a matrix of nickel. It is also responsible for the high corrosion resistance of alloys because it induces the formation of a stable passive layer. In addition, molybdenum

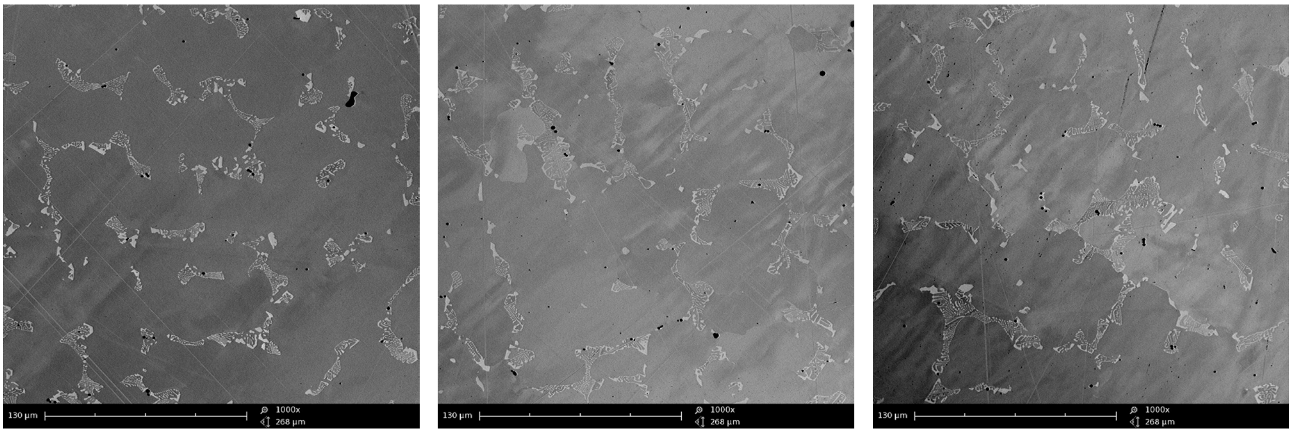

Figure 1. The microstructures of SEM alloys: (a) C1, (b) C2, and (c) C3

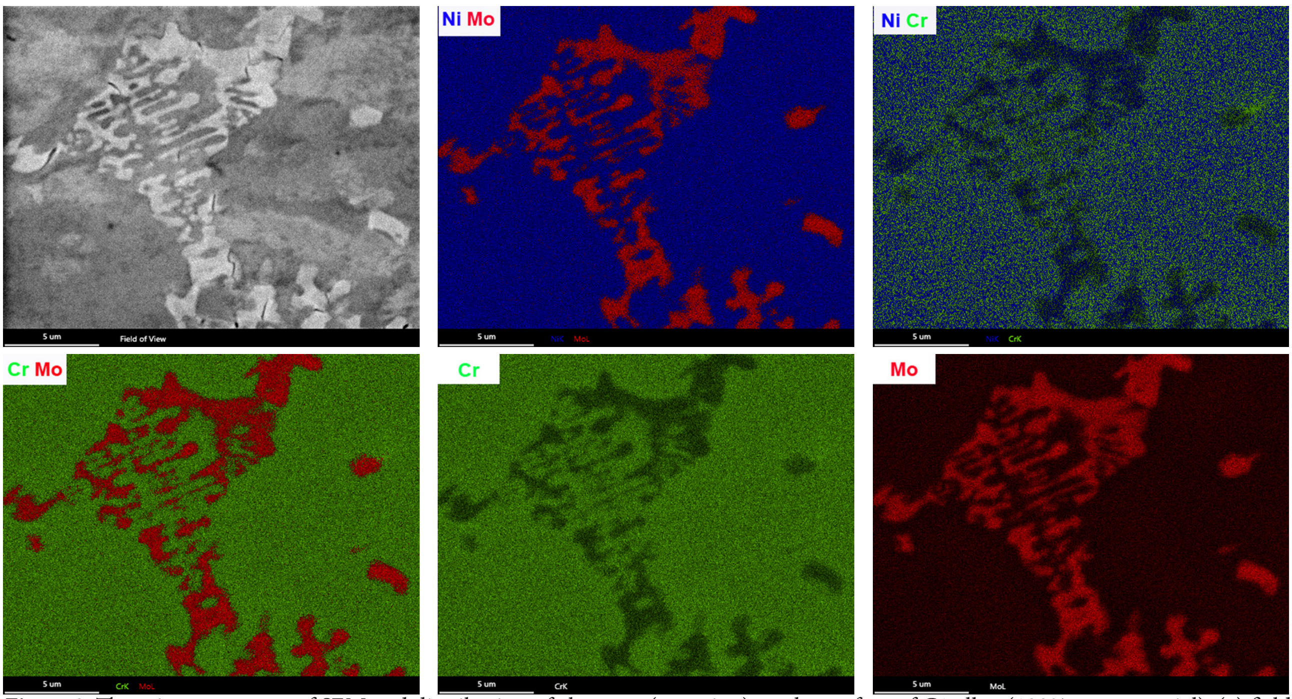

Figure 2. The microstructures of SEM and distribution of elements (mapping) on the surface of C1 alloy (100% new material): (a) field of view, (b) Ni and Mo, (c) Ni and Cr, (d) Cr and Mo, (e) Cr, (f) Mo

Table 2. Chemical composition of the tested NiCrMo alloys (masses %)

Alloy	C	Mn	P	S	Si	Cu	Cr	Mo	W
C1	0.066	<0.002	0.037	<0.002	~1.8	<0.002	24.45	10.49	0.064
SD	0.007	–	0.0025	–	–	–	0.259	0.101	0.005
C2	0.073	<0.002	0.035	<0.002	~1.8	0.0012	23.49	10.52	0.111
Sd.	0.018	–	0.0034	–	–	0.0005	0.312	0.258	0.087
C3	0.087	<0.002	0.035	<0.002	~1.8	<0.001	24.34	10.70	0.071
SD	0.0007	–	0.001	–	–	–	0.207	0.354	0.002
Manufactures	–	–	–	–	1.5	–	26	11	–
Alloy	**Fe**	**Al**	**Co**	**Mg**	**Nb**	**Ti**	**Sn**	**N**	**Ni**
C1	1.045	0.098	0.173	<0.001	0.0059	0.0058	0.0045	<0.005	61.75
SD	0.036	0.07	0.0025	–	0.0019	0.0005	0.0003	–	0.042
C2	1.086	0.272	0.197	0.0018	0.0074	0.0065	0.0049	0.012	62.37
Sd.	0.06	0.212	0.022	0.0014	0.0043	0.0008	0.0006	0.016	0.939
C3	1.030	0.0062	0.185	<0.001	0.0084	0.0051	0.0043	<0.005	61.70
SD	0.024	0.0017	0.0021	–	0.0004	0.0001	0.0001	–	0.225
Manufactures	<1	–	<1	–	–	–	–	–	61

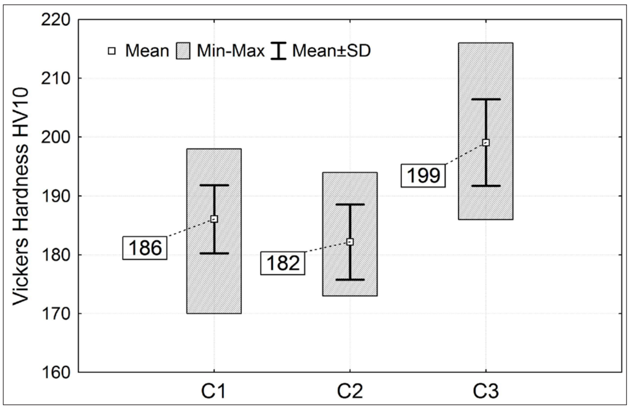

Figure 3. A comparison of the Vickers hardness of alloys studied groups Remanium CS + (N = 40)

is added because it increases resistance to pitting and crevice corrosion. Alloys with low content of Cr and Mo are, hence, more susceptible to corrosion [2].

Bauer *et al*. [18] claimed that the presence of carbon in the alloy promotes the reactions of certain alloying elements such as nickel, chromium, molybdenum, titanium and niobium. In turn, the presence of Ti in the alloy may affect the formation of more carbides and nitrides, because Ti is very reactive to carbon, and this increases the mechanical properties of alloys [18]. However, XRD analysis further discussed in publication [6] did not confirm phases of this type in the tested alloys.

Al-Hiyasat and Darmani [14] conducted tests on three alloys based on nickel (Remanium CS, Wiron 99 and CB Sof) with variable content of Cr, Mo and Cu. They demonstrated that casting has an impact on the release of elements included in the tested alloys. Here, casting increased the release of Cu and Fe, slightly less Co and Al, but in the case of Ni, the impact of casting was the lowest.

However, Yavuz *et al*. [15] observed significant differences in the content of Ni and Cr when re-remelting 6 different casting NiCr alloys (Kera N, Nodelco, Bellabond, Wiron 99, Metaplus VK and Tritech D) and pointed out that recasting adversely affects the quality of surface of tested materials.

Anusavice [19] indicated that elemental composition of NiCr alloy changes during the melting process. The results of these studies also demonstrated that mixing new and previously castings metal affects the stability of the chemical composition of NiCr alloy, which leads to changes in the quantity of main elements: Ni and Cr and other alloy additives such as Mo, Si, and Mn.

Presswood [20] came to different conclusions. He studied the chemical composition of NiCrBe alloys entirely recast up to four generations and noted that the composition of the alloy as a result of recastings does not change and was at a constant similar level.

In our work, the average hardness of new factory C1 alloy and an alloy with 50% addition of cast one C2 material reaches a value slightly lower than that declared by the manufacturer 195 HV10 (Fig. 3). However, for the C3 group, the average hardness value was slightly higher than that declared by the manufacturer.

Hardness test results were analysed by applying the Kruskal-Wallis test at the adopted level of significance $\alpha = 0.05$. However, no significant difference was found between C1 and C2 groups ($p = 0,143$), but significant differences were found between the C1 and C3 groups ($p < 0.05$) and between C2 and C3 ($p < 0.05$). Furthermore, the Mann-Whitney U test (for $\alpha = 0.05$), which is considered to be stronger than the Kruskal-Wallis test showed significant differences between all research groups ($p < 0.05$). The observed differences in hardness are closely related to the change in chemical composition and different types of secreted phases that occur during alloy recasting.

In contrast, the results of the research of Palaskar [21] show that there is no statistically significant difference between the hardness of new alloy and recast alloy. He also said that the re-cast of dental casting NiCr alloy is very beneficial both economically and environmentally.

In turn, Prabhu *et al*. [22] evaluated the mechanical properties (including micro-hardness) of new factory NiCr alloy and with the addition of 50% of recast material and recasted in its entirety. They saw significant changes between groups of tested alloys. Here, the authors found that the use of casting without the addition of 50% new alloy brought about adverse effects, in comparison to alloys composed of 100% new material.

Figure 4 reveals changes in bond strength of metal-ceramics carried out according to ISO 9693 for Remanium CS+ alloy. All the results obtained in this test meet the requirements for minimum strength $\tau b \geq 25$ MPa. Moreover, there was no deterioration in the adhesion of metal-ceramics for alloys with the addition of recast material. All analysed research groups also have a similar average adhesion at 48,51÷49,24 MPa. Here, slight differences in average values of adhesion are related to hardness of the base – the higher metal hardness of the base brings about greater rigidity of metal-ceramics. A statistical analysis by Kruskal-Wallis test and Mann-Whitney U test (for α=0.05) showed no statistically significant differences in the obtained results. These results lead to the conclusion that adding 50% recast material (casted once and twice) does not affect the adhesion of dental porcelain to the NiCrMo base.

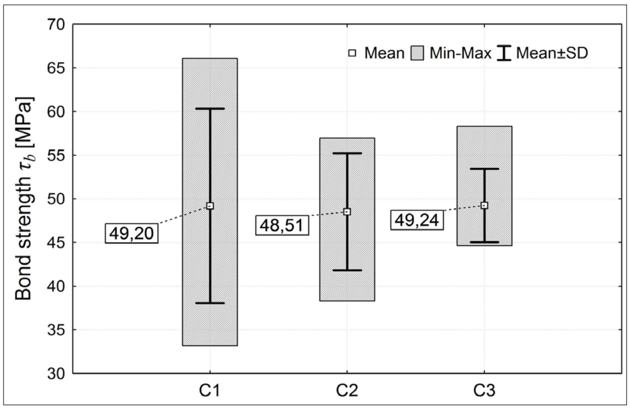

Figure 4. Summary of results of bond strength of metal-ceramic Remanium CS + alloys involving materials after recasting – results of three-point bending test conducted according to ISO 9693 (N = 9)

of post-production materials, at least 25% new material must be used.

In opposition, Madani *et al*. [24] on examining the impact of recast material on the metal-ceramics bond of NiCrBe alloys (Super Cast and Verabond) stated that the addition of 50% of recast material significantly reduces the bond strength of metal-ceramics.

What is more, in comparing the results of adhesion testing of ceramic-metal systems for new factory NiCrMo alloy with results presented in literature data, Yilmaz and Dinçer [3] obtained for Remanium CS alloy combined with ceramics 68 VMK (Vita, Germany) strengths in the three-point bending test of 46.6 MPa.

Figure 5 shows SEM images of cross-sectional views of transverse specimens after the test of adhesion of dental porcelain to the metal base of NiCrMo. In almost all groups of tested alloys, cohesive fractures were evident on the side of the metal base. Here, cracks occurred primarily in the area of the ceramics or in the boundary layer of metal oxides. This effect indicates the very good adhesion of NiCrMo

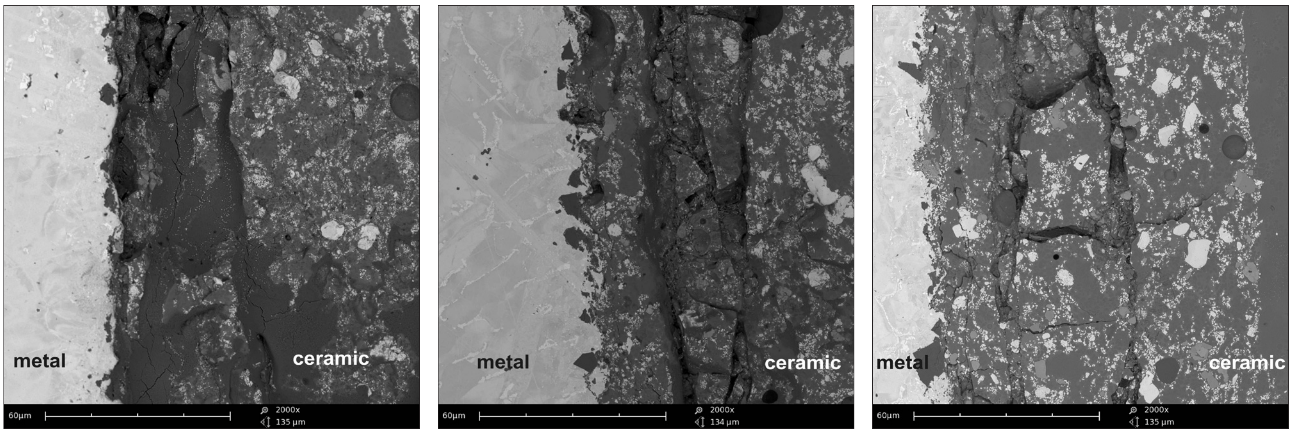

Figure 5. Microscopic SEM image of cross sections of metal-ceramics after adhesion testing for: (a) C1 castings made in 100% from the new factory material, (b) C2 castings made with 50% addition of recast material – cast once, (C) C3 castings made with 50% addition of recast material – cast twice.

Ucar *et al*. [16] suggest that veneering with ceramic of recast metals can cause a change in the composition of the metal oxide on the surface, which may be critical for the binding strength between the metal and ceramic material. The carried out research on adhesion of porcelain to NiCrMo alloys (Remanium CSe) has shown that the use of recast base metal alloy with a new alloy does reduce bond strength between metal alloy and ceramic material. In contrast, they obtained no statistically significant difference in bond strength of metal-ceramic alloys involving recast material.

In turn, Madahav *et al*. [23] by means of three-point bending tests compared results from the use of new factory alloys and alloys with different percentages of recast materials on the adhesion of porcelain to nickel-chromium alloy. Within the framework of these experiences, the authors demonstrated that factory new alloys have the highest adhesion, but they did not noticed any significant changes in bond strength of porcelain for alloys with up to 75% re-used recast material. However, they argued that in order to gain permanent bond of NiCr alloy and porcelain in the case base to dental porcelain. During ISO 9693 tests for all tested samples upon exceeding the value of force F_{fail}, the porcelain did not fall off completely from the base. Moreover, loss of adhesion of metal-ceramic was accompanied by an acoustic effect in the form of audible cracks of ceramics and a decrease in the value of force F_{fail}.

SUMMARY

Use of an additive material from multiple recasts to NiCrMo alloy results in the following changes:

The addition of 50% of recast material causes a slight increase in the concentration of C% with the number of repeated NiCrMo castings and differences in the chemical composition of Mo and Cr, compared to what declared by the manufacturer.

The addition of 50% of NiCrMo alloy brings about a change in phase composition, contributing to the creation of new intermetallic phases.

The addition of 50% of double-melted material engenders a statistically significant increase in the hardness of NiCrMo alloy.

There was no deterioration in the adhesion of metal-ceramic NiCrMo alloys involving recast material, and the obtained results are maintained at a constant high level – meeting the requirements of ISO 9693.

On the basis of own research, it has been found that it is possible to get durable materials used to produce prosthetic apparatuses in which at least 50% of the material is brand new material, and the remaining part of the alloy may come from single or double recasts. However, in this study, we tested alloys with an addition of 50% of recast material only up to the level of second generation. Therefore, the authors suggest recasting of alloy additive to the level of the specified number of generation and testing the influence of the higher level of number of recast on the properties of the alloy. The use of recasting procedures can reduce costs of NiCrMo castings, and conducted tests indicate that it may be safe in dentistry. The alloys obtained by the authors with the addition of recast materials meet the quality requirements for materials for the manufacture of prosthetic apparatuses relating to their quality and durability, and therefore can be used in dental laboratories.

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