

## EFFECT OF RECASTING ON THE STRUCTURE AND PROPERTIES OF COMMERCIAL Ni-Cr DENTAL ALLOY

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**Abstract:** *The Ni-Cr dental alloys are among the oldest restorative materials used in dentistry. Reuse of previously melted and cast dental alloys is a routine procedure used in dental laboratories to reduce the cost of dental restorations. Continuous reuse of the commercial Ni-Cr dental alloys, such as Wirron 99, can change numerous properties of these materials, and therefore the present study was aimed to establish the outcome of several recasting cycles on the Wirron 99 alloy properties. Obtained results reveal that alloy recasting resulted in the appearance of typical dendritic microstructures where the chemical composition of dendritic and interdendritic regions differs. Moreover, the results of the present study showed that the number of recasting cycles has a significant effect on the alloy microstructure, structure, electrical conductivity, and hardness.*

**Keywords:** *Ni-Cr dental alloy, microstructure, electrical conductivity, micro- and macrohardness*

### 1. Introduction

Metallic materials (mainly alloys) are widely used in dental surgery for the production of most of the surgical instruments for face and jaw surgery, as well as in restorative dentistry, orthodontics, and dental prosthetics as a building material. Usage of dental alloys has a long tradition in restorative dentistry, but their application in the true sense of the word starts at the beginning of the 20th century with the introduction of the casting apparatus based on the centrifugal force principle [1,2]. The development of dental alloys followed the development of metallurgy with respect to the main requirements of metallic materials biomedical usage. Namely, one of the main requirements for dental metallic materials usage is high corrosion resistance of these materials in the human body environment, such as the oral cavity [1,3,4].

Dental casting alloys can be categorized in several different ways, but the classification system mostly used by dental practitioners is the American Dental Association (ADA) compositional classification system. The ADA classification system, established in the year 1984, classifies casting alloys into three main groups based on their wt.% compositions [1] as follows:

- a. high-noble (HN) alloys with an overall noble metals content (sum of gold, palladium, and platinum content) of at least 60 wt.% and gold content of at least 40 wt.%,
- b. noble (N) alloys with at least 25 wt.% of noble metals content with no specific requirements for gold content in the alloy composition, and
- c. predominantly based (PB) alloys with less than 25 wt.% of noble metals content in the alloy composition with no additional compositional specifications.

Today's world market offers a large number of dental alloys while the modern dental industry strives to create new alloys that would be biocompatible and chemically resistant in a corrosive environment while possessing good mechanical properties. The ideal dental alloy does not exist in today's market since the dental alloy manufacturers are trying to produce an alloy for every budget [5,6]. In developed countries, such as the United States of America, European Union countries, and Japan, cast gold alloy and all-ceramic materials are the most commonly used in dental practice. On the other hand, in developing countries, the base alloys (Ni-Cr and Co-Cr alloys) and prefabricated stainless-steel crowns are the most prevalent types of used metallic dental materials [6]. It should be additionally emphasized that approximately 90% of all removable partial dentures used all over the world are nowadays cast from the non-precious alloys that contain Co, Cr, and Ni [7]. However, in

less economically developed countries (LEDCs), the reuse of previously melted and casted alloys is a routine procedure used in dental laboratories to further reduce the cost of dental restorations [8]. The available literature in this field [8-13] indicates that this procedure is widely used in everyday practice, despite the clinical suggestion that such a procedure should be avoided since the content of new and previously unused dental alloy in the overall dental alloy castings must be at least 50% in order to avoid an increase of the cytotoxicity of Co-Cr, Ni-Cr, and Au-Pt alloys [8]. Higher content of the already remelted alloy then suggested is however used in the case of the noble alloys [11,14-16], as well as in the case of the basic dental alloys [17-22]. The previous literature reports show that the chemical composition of the dental alloys changes with their repeated remelting and, as a consequence, reduction of the content of alloying elements, such as Ni, Cr, Cu, Sn, Zn, Cr, Ti [8,15], and Fe due to evaporation and oxidation [15], can be expected. Mentioned reduction of the content of these elements can significantly weaken the metal-ceramics bond [17]. Furthermore, it was also shown that the repeated remelting influences a change in the alloy grain size [11], the content of impurities and microporosity [11], and the change in microstructure and mechanical properties [8,11,23]. In general, new and reused dental alloys do not have the same composition, microstructure, physical and mechanical properties, color, corrosion resistance, ions release tendency, and most importantly, they do not have the same biological quality [24].

This study, therefore, aims to establish the outcome of repeated alloy recasting on the microstructure, structure, electrical conductivity, and hardness of commercially attainable nickel-chromium alloy, *i.e.* Wirron 99.

## 2. Experimental Procedures

Commercially available Ni-Cr-based dental alloy Wirron 99 (Bego, Germany) was used in this study. The alloy composition is shown in Table 1, as provided by the alloy manufacturer.

**Table 1.** Chemical composition of the commercial Wirron 99 alloy (in wt.%).

Alloy type	Ni	Cr	Mo	Nb	Si	Fe	Ce	C
Ni-Cr	65.00	22.50	9.50	1.00	1.00	0.50	0.50	0.02

Disk-shaped Wirron 99 samples with 6 mm in diameter and thickness of 1 mm were cast in an air atmosphere using the laboratory induction furnace at the Clinic for Dental Prosthetics of the Faculty of Dental Medicine, University of Belgrade. The casting of the alloy was performed at 1420 °C, according to the manufacturer's specifications. Alloy castings were examined after a different number of repeated casting cycles, *i.e.* first (sample W1), fourth (sample W4), and eighth (sample W8). Each casting cycle consisted of subsequent melting and casting sequences.

### 2.1 Microstructural characterization

For microstructural analysis, the casted alloy samples were wet ground and polished using the standard metallographic preparation procedure. Alloy samples were afterward cleaned with alcohol and distilled water and air-dried. The disc-shaped alloy castings were additionally etched using the etching solution that contains 10 ml HNO<sub>3</sub> and 40 ml HCl [25,26]. The polished and etched samples were observed under a Carl Zeiss Axiovert 25 light optical microscope (LOM) equipped with the digital Panasonic WV-CD50 camera, and an FEI Quanta 200 scanning electron microscope (SEM) equipped with an Oxford Instruments INCA X-sight energy dispersive spectroscope (EDS).

The x-ray diffraction (XRD) patterns were obtained from the mechanically polished alloy samples using a Siemens D5000 PC automatic diffractometer with Cu K $\alpha$  radiation. The XRD patterns were recorded in the 2 $\theta$  range from 40° to 80° with a scanning rate of 0.02°/s.

### 2.2 Hardness testing and electrical conductivity measurements

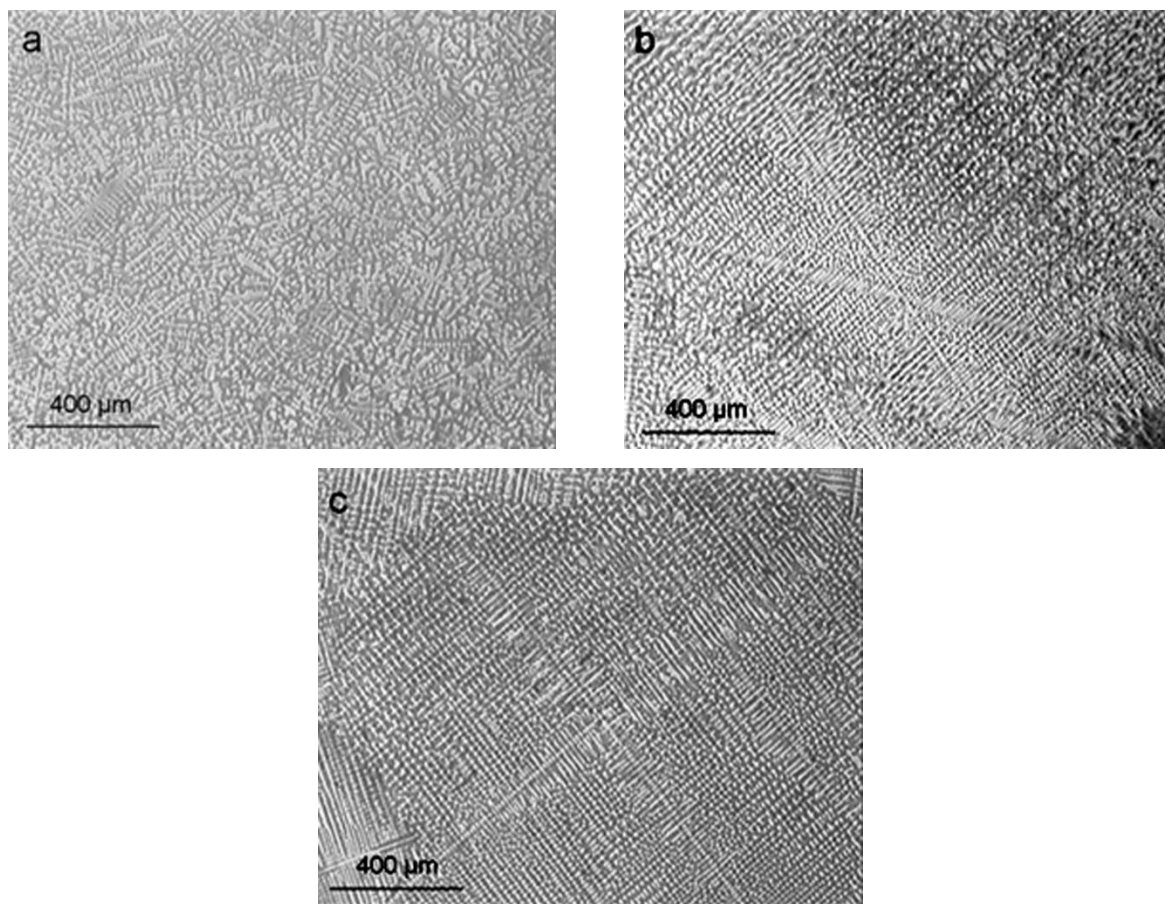
The macrohardness and microhardness measurements of the casted alloy samples were performed using a Buehler SemiMacro Vickers 1112 HV112 and a Buehler MicroMet 5101 Vickers

hardness tester with a load of 1 kgf (9.805 N) applied for 5 s and 25 gf (245.2 mN) applied for 10 s, respectively. Results were obtained as the average values of five consecutive measurements.

The electrical conductivity of the casted alloy samples was measured using a Forested SIGMATEST 2.069 conductivity meter [27]. A frequency of 960 kHz was applied during measurements due to the small sample thickness. Obtained results were presented as an average value of fifteen consecutive measurements.

### 3. Results and Discussion

Fig. 1(a-c) shows the typical dendritic microstructures of the recast Ni-Cr alloy samples. It is well known that a dendritic structure usually exhibits compositional variations, with the dendrite arms containing fewer alloying and impurity elements than interdendritic regions. Because of such compositional changes, the rate of etching at interdendritic regions differs from that at dendrite arms [15]. Microstructural changes, induced by an increase in the number of recasting cycles, could be observed in the alloy microstructure even at small magnification.

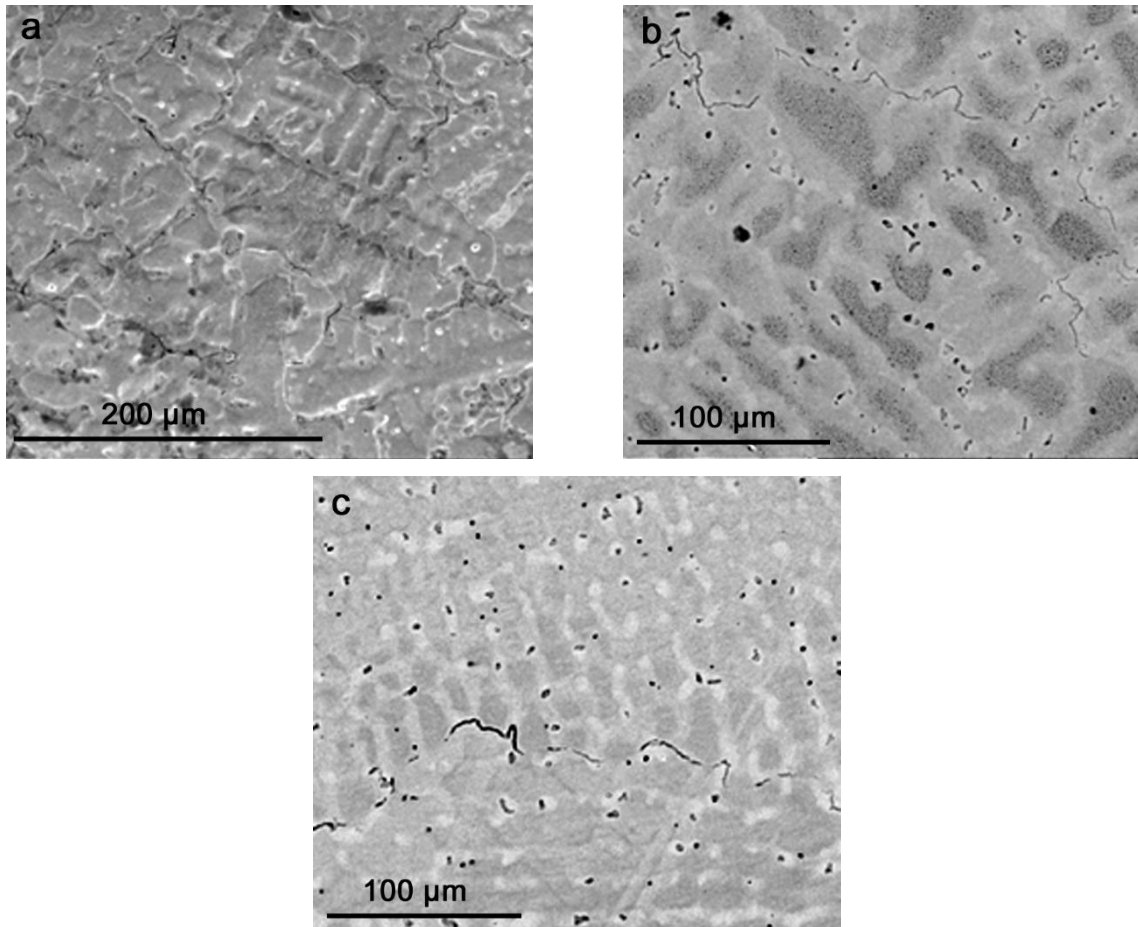


**Figure 1.** LOM micrographs showing the typical microstructure of Ni-Cr dental alloy after (a) first, (b) fourth, and (c) eighth recast cycle.

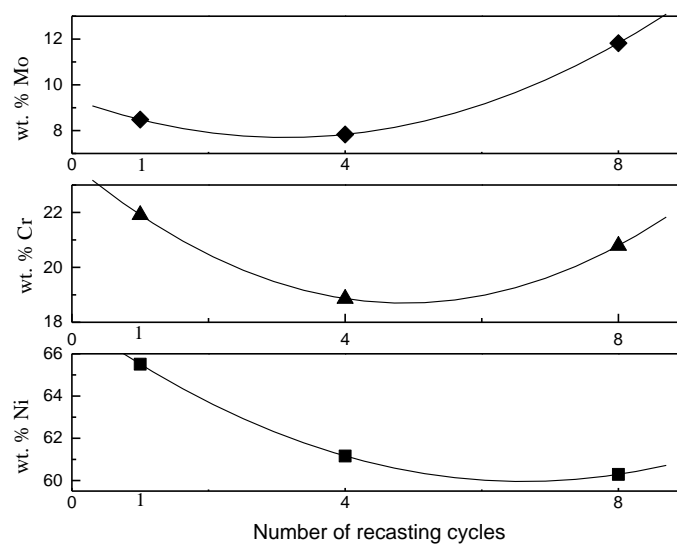
SEM microphotographs showing the microstructure of Ni-Cr alloy after the first, fourth, and eighth recast cycle are given in Fig. 2(a-c). The dendrite structure is clearly observed. With the increasing number of melting and casting sequences pronounced crystal segregation appears (Figs. 2b and 2c). In the complex system, such as dental Wirron 99 alloy, in addition to the solid solution, phases in the interdendritic region of irregular shape appeared. The presence of precipitates, which are probably Mo- and Nb-carbides, can also be observed along the grain boundaries [15].

EDS analysis of the dendritic and interdendritic regions did not indicate significant differences in the chemical composition between these zones, and because of that, a semi-quantitative surface analysis was performed. Based on the obtained EDS information, shown in Fig. 2(a-c),

variation of the primary elements (Ni, Cr, and Mo) content in the alloy was presented in Fig. 3. With an increase in the recasting cycle number, the content of Ni decreases from 65 wt.% up to approximately 60 wt.%. Quite the opposite trend was observed for the change in Mo content.

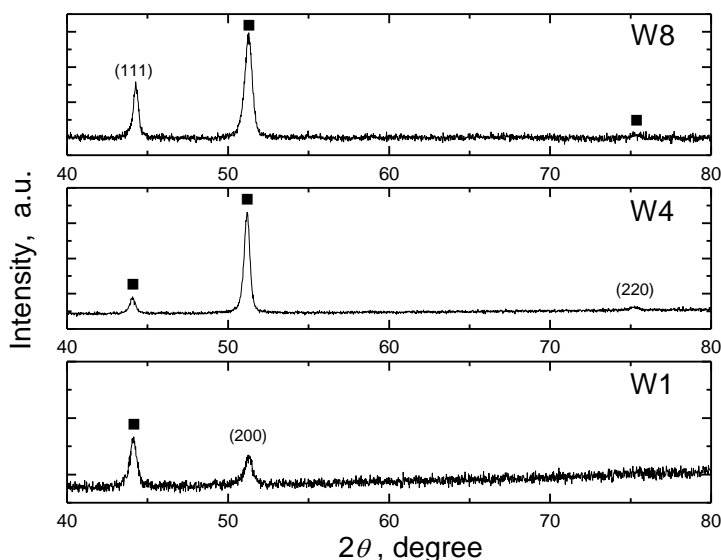


**Figure 2.** SEM micrographs showing the microstructure of Ni-Cr dental alloy after (a) first, (b) fourth, and (c) eighth recast cycle.

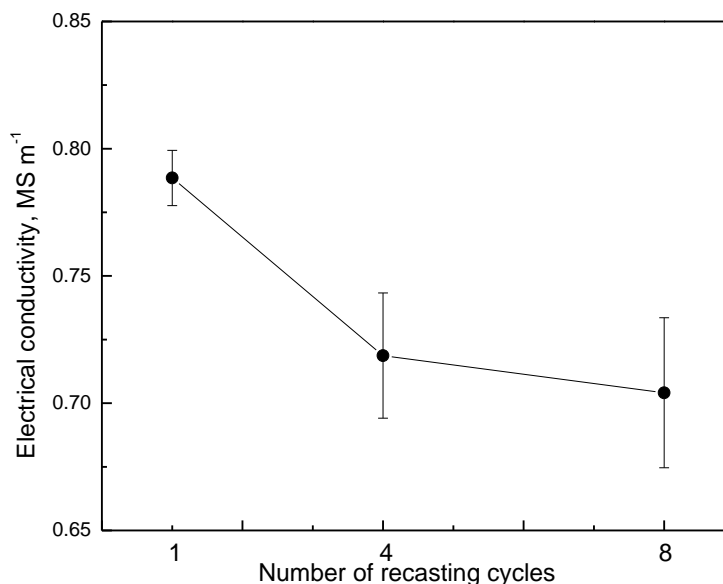


**Figure 3.** Variation of the primary elements (Ni, Co, Mo) content in the Wirron 99 alloy with an increase in the recasting cycle number.

XRD patterns of the Ni-Cr alloy after the first (W1), fourth (W4), and eighth (W8) melting and casting sequences are shown in Fig. 4. Only the characteristic peaks of  $\alpha$ -Ni phase (■), as the Ni-rich solid solution with face-centered cubic (fcc) lattice, may be seen. With an increase in the number of repeated casting cycles, a change in the intensity of peaks from  $(111)_{fcc}$ ,  $(200)_{fcc}$ , and  $(220)_{fcc}$  planes can be observed. Moreover, the preferential orientation has changed from  $(111)_{fcc}$  to  $(200)_{fcc}$  planes. The reason for this behavior can be found in the alloy chemical composition variation during the successive melting and casting sequences, *i.e.* fitting alloying elements in precisely defined planes.



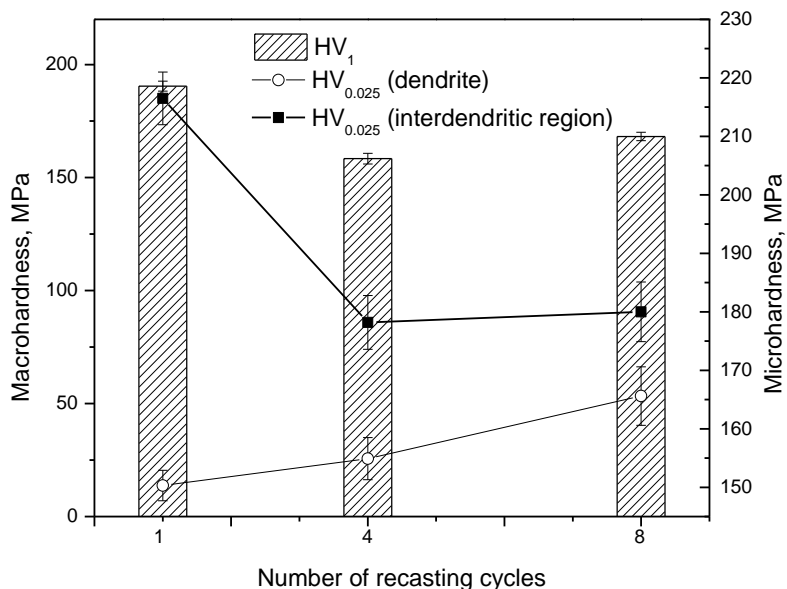
**Figure 4.** XRD patterns of the Ni-Cr dental alloy after (a) first (W1), (b) fourth (W4), and (c) eight (W8) recasting cycle.



**Figure 5.** Electrical conductivity of multiple casting samples of the Ni-Cr dental alloy.

The information about the electrical conductivity of most of the materials used in dentistry is limited in the available literature. Starčuková et al. [5] reported that the difference in the electrical conductivity of 45 metallic dental materials is in the range from 0.8 MS m<sup>-1</sup> to 9.41 MS m<sup>-1</sup>. Based on their results, the Ni-Cr alloys are in the group of dental alloys with the lowest value of electrical conductivity ranging from 0.80 to 0.89 MS m<sup>-1</sup>, and the Wirron 99 alloy has a value of 0.8 MS m<sup>-1</sup>. The electrical conductivity of Ni-Cr alloys was found to be low and in the narrow range of 0.7-0.78 MS m<sup>-1</sup>. The values of electrical conductivity of the recast samples of Ni-Cr-based dental alloy

indicate that when the number of recasting cycles increases the concentration of impurities, such as oxides, nitrides, and carbides, causes a decrease in electrical conductivity (Fig. 5). Furthermore, it was reported that several alloying elements in the Ni-Cr alloys (Mo, Nb, and Fe) can form oxides and nitrides at high temperatures with gases from the surrounding atmosphere that can alter the properties of these alloys [28].



**Figure 6.** Effect of the recasting cycle number on the macrohardness and microhardness of dendritic and interdendritic regions in the Ni-Cr dental alloy.

The correlation between the number of recasting cycles and the macro- and microhardness of dendritic and interdendritic regions of the alloy is shown in Fig. 6. It can be noticed that with the recasting cycles number increase the macro- and microhardness of interdendritic regions decrease with a slight discrepancy observed for samples obtained after the fourth and eighth recasting. On the other hand, the microhardness of dendrites increases with the recasting cycle number increase. The microhardness of interdendritic regions is higher than that of dendritic areas. The difference between the microhardness of these two regions (dendritic and interdendritic) is decreased with an increase in the recasting cycle number and tends to equalize. The minor difference in the concentration of elements between these two zones is additionally confirmed by the slight difference in the level of microhardness that decreases in series 66→22→15 MPa with the recasting cycle number increase.

#### 4. Conclusions

Commercially available Ni-Cr-based dental alloy Wirron 99 was investigated to determine the effect of multiple recasting processes on the alloy structure and properties. For this purpose, the number of recasting cycles was selected to be one, four, and eight. The alloy microstructural and structural alterations were examined in detail. All tested samples showed dendritic solidification microstructure that changes with the number of recasting cycles. An increase in the recasting cycle number resulted in a decrease in the interdendritic regions' hardness and an increase in the dendrites' hardness. The electrical conductivity of the recast Wirron 99 alloy decreased with an increase in the number of recasting cycles due to an increase in the impurities concentration.

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