



EFFECT OF METAL PRIMER ON THE SHEAR BOND STRENGTH OF POLY METHYL METHACRYLATE HEAT CURED DENTURE BASE RESIN TO CAST TITANIUM AND COBALT-CHROMIUM ALLOYS: AN IN-VITRO STUDY

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ABSTRACT

Background: Poor chemical bonding of acrylic resins to metal alloys can result in microleakage and failure of the bond. Metal primer has been shown to be effective in improving the bond strength of acrylic resins to metal alloys. However, there is insufficient information about their effect on bonding to different types of metals. **Aim and Objective:** The purpose of this study was to evaluate the effect of metal primer on the shear bond strength of PMMA heat cured denture base acrylic resins to cast Titanium and Cobalt-Chromium alloys. **Material and methods:** A total of 40 disk-shaped wax patterns (10 mm in diameter and 3 mm thick) were cast in Titanium (Tritan) and Cobalt-Chromium alloy, (Remanium G 800). After casting, the disk surfaces were finished till 600 grit abrasive paper under water. The specimens were numbered and then divided into 4 groups (n=10). Samples were subjected to airborne particle abrasion with aluminum oxide 250 μ m at 4.5 kgf/cm² pressure. The distance between the nozzle tip and the specimen surfaces was maintained at 5 mm, and sandblasted for 5 seconds and then ultrasonically cleaned in ultrasonic cleaner for 10 min in acetone and air dried to ensure the removal of all residual particles and surface contaminants. The specimens were divided into 2 Groups for each metal (n=10) which were air abraded with 250 μ m aluminum oxide and the other 2 groups for each metal (n=10) which were air abraded with 250 μ m aluminum oxide and then received the Alloy Primer. The specimens were flaked and Lucitone 199 PMMA denture base resin was used to make the resin tags. The specimens were tested in a universal testing machine at a crosshead speed of 0.5 mm/min in shear mode till the resin fractured from the metal surface. Stereo zoom microscope was used to study the mode of failure between the metal and resin. Data (MPa) were analyzed using Paired t- test with significance level (p=0.05). **Results:** The paired t- test indicated that shear bond strength (SBS) values varied according to the surface preparations used ($P<0.001$). The SBS between Titanium and heat-polymerized resin (Group II) with sandblasted and Alloy Primer applied was the highest (2.73 MPa), and the SBS between Cobalt-Chromium and acrylic resin (Group III) which was only air abraded, was the lowest (1.93 MPa). The SBS values between sandblaste (Group I & III) and sandblasted and primed specimens of Titanium and Cobalt-Chromium alloys, (Group III & IV) respectively showed significant difference ($p<0.001$). When comparing the bond strength of sandblasted and Primed Titanium (Group II) with sandblasted and primed Cobalt-Chromium (Group IV), Titanium (Group II) showed the highest bond strength. Adhesive type of bond failure was noted with air abraded specimens where in, mixed type of bond failure was noted with air abraded and primed specimens. **Conclusion:** The metal primer was associated with an increase in the adhesive bonding of acrylic resins to irrespective of the metal alloys. The shear bond strength of heat cured acrylic resin to the Titanium alloy was significantly higher than to that of Cobalt-Chromium alloy.

Keywords :- Cobalt-chromium alloy, Denture base resin, Shear strength, Titanium alloy.

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INTRODUCTION

In Prosthodontics, removable partial dentures (RPD) are commonly fabricated with acrylic resin and metal. Metals like cobalt-chromium (Co-Cr alloy) or commercially pure titanium (CP Ti) are commonly used for removable partial denture's that contain metal frameworks, bars, or clasps. Such metals are well suited for frameworks because of their mechanical properties, biocompatibility and corrosion resistance. Heat polymerized acrylic resin is also generally utilized as a denture base polymer for removable prosthesis. The bonding between the metal components and the denture base resin plays an important role in the longevity of the prosthesis. Deficiencies in bonding at the metal-resin interface can become a significant clinical problem, leading to failure of the prosthesis [1].

Cobalt-chromium (Co-Cr) alloys are frequently used to fabricate denture frameworks due to their favorable mechanical properties, whereas commercially pure titanium (CP Ti) and titanium alloys are preferred for their biocompatibility as well as desirable mechanical properties.

To eliminate some of these problems associated with Cobalt-Chromium alloys, titanium has been increasingly used for RPD frameworks.

The mechanical retention for a denture resin poly methyl methacrylate, (PMMA) in removable prostheses is usually provided by the framework design in the denture base, such as through the use of beads, posts, an open lattice, a mesh, or some other macroscopic retentive design. External and internal finishing lines should be placed on the cast metal framework wherever the acrylic resin joins the cast framework. If there is a separation between the acrylic resin and the cast metal, especially at the finishing line, cracks or crazing may occur in the acrylic resin, leading to microleakage that is accompanied by staining. Microleakage from the metal-PMMA interface can lead to discoloration, deterioration of the resin, and the creation of a reservoir for oral debris and microorganisms. Incomplete fracture or total separation of the resin can also occur. The lack of a chemical bond can directly affect the metal-resin interface. The difference in the coefficients of thermal expansion between the metal and the resin might create a gap at the interface, leading to microleakage. Therefore, conventional adaptation between the acrylic resin denture base and the metal framework may not be sufficient to prevent microleakage [2]. Significant research has focused on improving the chemical bond strength between the acrylic resin and the metal to withstand intermittent occlusal forces and endure the constant moisture from saliva and temperature variations in the oral environment [3].

Various methods and techniques such as silica coating, chemical etchants, spark erosion, and tinplating have been tried in order to enhance bonding of the resin to

the metal. The bonding system of an adhesive primer containing functional monomer is considered a technique that facilitates chemical adhesion. Chemical bonding is more desirable than mechanical retention when metal is incorporated. Compared with traditional methods, the primer system is simpler, easier and more economical because it doesn't require special equipment and is not technique sensitive. Primers containing 10-methacryloxydecyl dihydrogen phosphate (MDP) have been successfully used to enhance the bonding of resin to base metal alloys. Although a number of reports are available concerning the bonding of autopolymerized resin, composite resin, or luting agents, little information is available on bonding a heat-polymerized denture base resin to Titanium with priming agents. This study was designed to evaluate the effect of priming agents on the bond durability between Titanium and Cobalt-Chromium alloy and a heat polymerized denture base resin. It was hypothesized that the bond durability would be enhanced by the priming agents [1].

The purpose of this study is to determine the effect of metal primer on the shear bond strength of poly methyl methacrylate heat cured denture base resin to cast titanium and that of cobalt-chromium alloys.

MATERIALS AND METHODS

This is an in – vitro study and sample size is 40. The samples were grouped as following category

1. Group I- Ti + Sandblast. [n=10]
2. Group II – Ti + Sandblast + Alloy primer. [n=10]
3. Group III – Co-Cr + Sandblast. [n=10]
4. Group IV – Co-Cr + Sandblast + Alloy primer. [n=10]

Fabrication of Titanium Samples

For Group I & II, twenty commercially pure (CP) titanium cast metal discs were prepared using a mould of 10 mm length and 2.5 mm width in the center. Wax patterns were prepared by flowing molten casting inlay wax (Renfert, Germany) into the square-shaped mould space with the help of a thermostat (Dentaurum).

Five wax patterns were attached at a time to the runner bar sprue former. It was then fixed to a 9 size crucible former (Titec-Orotig, Verona, Italy) [Fig 1] and the wax patterns were sprayed with a surfactant liquid to reduce the surface tension (Picosilk, Renfert, Germany) and blow dried. A moistened, asbestos-free ring liner (Kera Vlies, Dentaurum, Germany), 4 mm short at either end of the ring, was attached to the crucible former and the wax patterns were vacuum invested (Mestra, Germany) at 26-29" Hg(660-740 mm) pressure using 500 g of magnesia based spinel investment material [$MgAl_2O_4$] and 75 ml mixing liquid (Titec- Orotig, Verona, Italy) as per manufacturer's instructions.

After setting of the investment, the crucible former was separated and the ring was placed inside the wax burnout furnace (Bego, Germany)

The invested casting ring was heated in this furnace according to the following schedule

1. 55 °C/min increase in temperature until it reached 150 °C, with a 90 min dwell (holding time)
2. 5° C/min until it reached 250 °C, with a 90 min dwell at this temperature
3. 5 °C/min until it reached 950 °C, with a 60 min dwell time at this temperature [4].

After reaching the maximum temperature of 950 °C, the casting ring was cooled to 450 °C gradually and casted immediately in an automatic titanium casting machine (Type F-250, Orotig, Italy) using CpTi (Tritan, Dentarum, Germany) ingots. The casting machine automatically evacuated the chamber, which was filled by argon gas in 70 s. The argon supply continued for approximately 120 s after the molten metal had dropped into the mould. The machine then stopped automatically to allow air to enter the chamber [5].

Once cooled to room temperature, the specimens were devested and sandblasted with aluminium oxide powder (Renfert, Germany). The samples were standardized by holding it to a fixed plane unidirectionally on a horizontal grinding unit and polished with silicon-carbide abrasive paper 120–600 grit (Dentarum, Germany) in sequence and by using rotary equipment.

25 Samples were fabricated and analyzed visually for gross surface defects. From this, the sample size was brought to 20. Samples were then scrubbed under running water and then cleaned in an ultrasonic cleaner (Mestra, Germany) using distilled water.

Fabrication of Cobalt-Chromium samples

For group III & IV, twenty cobalt-chromium metal discs were prepared using a mould of 10 mm length and 2.5 mm width in the center. Wax patterns were prepared by flowing molten casting inlay wax (Renfert, Germany) into the square-shaped mould space with the help of a thermostat (Dentaurum).

Seven wax patterns were attached at a time to the runner bar sprue former. It was then fixed to a 9size crucible former (Orotig, Verona, Italy) and the wax patterns were sprayed with a surfactant liquid to reduce the surface tension (Picosilk, Renfert, Germany) and blow dried.

The samples were fixed to the crucible former and the wax patterns were vacuum invested using 26-29" Hg (660-740 mm) pressure with 500 g of Phosphate based investment material (Bellavest T; BEGO) and 75 ml mixing liquid (BegoSol; BEGO) as per manufacturer's instructions. After setting of the investment, the crucible former was separated and the ring was placed inside the wax burnout furnace (Bego, Germany). Cobalt-Chromium-Molybdenum alloy (Remanium GM800, Dentarum, Germany) was used to prepare specimens. After Burnout

procedure, using the Argon caster (Renfert, Germany) casting was done.

Once cooled to room temperature, the specimens were devested and sandblasted with aluminum oxide powder (Renfert, Germany). The samples were standardized by holding it to a fixed plane unidirectionally on a horizontal grinding unit and polished with silicon-carbide abrasive paper 120–600 grit (Dentarum, Germany) in sequence and by using rotary equipment.

25 Samples were fabricated and analyzed visually for gross surface defects. From this, the sample size was brought to 20. Samples were then scrubbed under running water and then cleaned in an ultrasonic cleaner (Mestra, Germany) using distilled water.

Standardized Sandblasting Procedure:

Samples were subjected to air borne particle abrasion with aluminium oxide 250 µm at 4.5 kgf/cm² pressure. The distance between the nozzle tip and the specimen surfaces was maintained at 5 mm, perpendicular to the tip and sandblasted for 5 seconds and then ultrasonically cleaned in ultrasonic cleaner (Mestra, Germany) for 10 min in acetone and air dried to ensure the removal of all residual particles and surface contaminants [6,7].

Mode of Bond Failure

Fractured test specimens were examined using Stereo Zoom Macroscope (Macscope-3, Samsung SDC-313B) at10X magnification for nature of bond failure between resin to metal interface and were classified as adhesive, cohesive and mixed type

Scanning Electron Microscope (SEM)

Two samples from each group were subjected to SEM analysis (SEM, Model JSM-6610 LV, Joel India Pvt. Ltd.). The surface preparation using sandblasting and primer application were subjected to SEM analysis under 500X magnification with a resolution of 20 µm and the accelerating voltage of 20 KV. Photomicrographs representing each group on the mode of surface preparation were prepared.

From the SBS values Paired t- test was used to analyze the data

($p \leq 0.05$) using SPSS software with mechanical and chemical surface treatments as independent variables.

RESULTS

The highest SBS of Titanium and PMMA resin was 2.39 MPa and the lowest was 1.52 MPa. The mean SBS was found to be 2.04 MPa.

The highest SBS of Titanium and PMMA resin was 3.21 MPa and the lowest was 2.29 MPa. The mean SBS was found to be 2.73 MPa.

The highest SBS of Titanium and PMMA resin was 2.29 MPa and the lowest was 1.52 MPa. The mean SBS was found to be 1.93 MPa.

The highest SBS of Titanium and PMMA resin was 3.15 MPa and the lowest was 1.98 MPa. The mean SBS was found to be 2.49 MPa.

When comparing the mean shear bond strength of Titanium (Group I) 2.04 MPa and Cobalt – Chromium (Group III) 1.93 MPa, there was no statistically significant difference ($p=0.331$).

When comparing the mean shear bond strength of Titanium (Group II) 2.72 MPa and Cobalt – Chromium (Group IV) 2.49 MPa there was statistically significant difference ($p=0.001$).

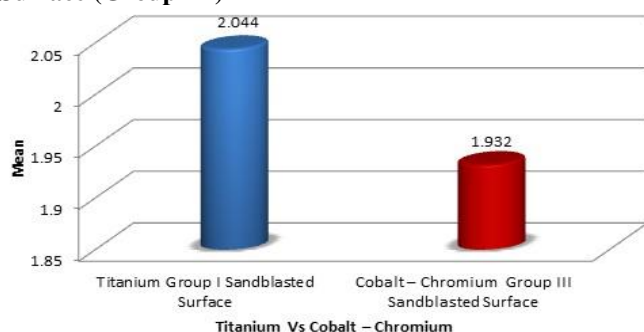
When comparing the mean shear bond strength of sandblasted Titanium (Group I) 2.04 MPa with sandblasted and primed Titanium (Group II) 2.72 MPa there was statistically significant difference ($p=0.001$).

When comparing the mean shear bond strength of Titanium (Group II) 2.72 MPa and Cobalt – Chromium (Group IV) 2.49 MPa there was statistically significant difference ($p=0.001$).

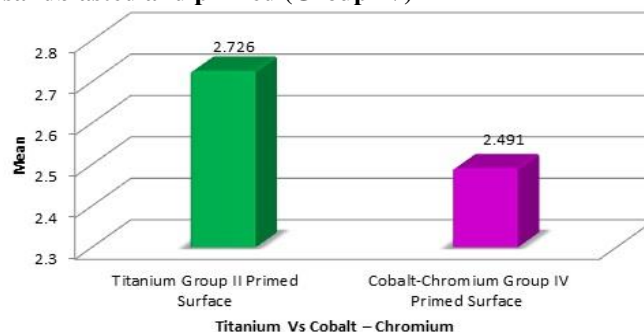
When comparing the mean shear bond strength of sandblasted

Cobalt – Chromium (Group III) 1.93 MPa with sandblasted and primed Cobalt – Chromium (Group IV) 2.49 MPa there was statistically significant difference ($p=0.001$).

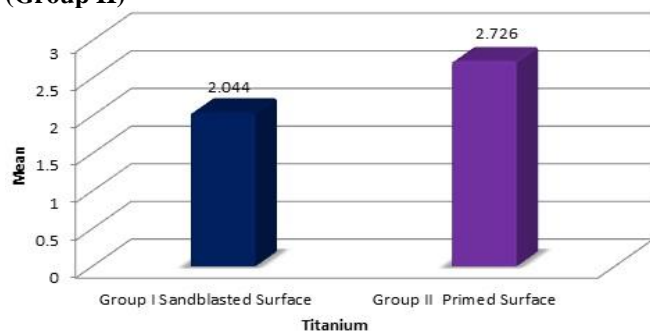
Graph – 1 Showing mean value for Titanium sandblasted Surface (Group I) Vs Cobalt – Chromium sandblasted Surface (Group III)



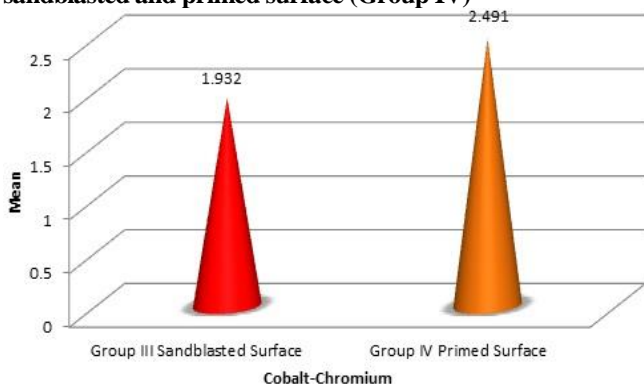
Graph – 2 Showing mean value for Titanium sandblasted and primed (Group II) Vs Cobalt – Chromium sandblasted and primed (Group IV)



Graph – 3 Showing mean value for Titanium sandblasted surface (Group I) Vs Titanium sandblasted and primed surface (Group II)



Graph – 4 Showing mean value for Cobalt- Chromium sandblasted surface (Group III) Vs Cobalt- Chromium sandblasted and primed surface (Group IV)



DISCUSSION

Co-Cr alloys have been used commonly for removable partial denture frameworks and also for complete dentures that incorporate metal components. The advantages of Co-Cr alloys are being relatively inexpensive and more rigid. However, Co-Cr alloy contains elements like nickel which might produce sensitivity or allergic reactions in some patients. Also, the Co-Cr frameworks are bulkier which causes discomfort to the patients [8].

Titanium was introduced in dentistry as implant material due to its excellent mechanical and physical properties and its use has been extended to fixed partial denture frameworks and now towards cast partial denture frameworks, owing to the advancement of casting procedures and materials. Titanium is relatively more biocompatible and light weight when compared to Co-Cr frameworks.

Cast partial dentures involve bonding of denture base resin to metal alloys. Failure of bonding between metal and resin can accumulate oral debris and

microorganisms which can lead to the failure of the prosthesis. So, the integrity of the bond at the resin-metal interface is important for the service longevity of the prosthesis [7].

Various methods had been introduced for improving the bond strength between acrylic resin and removable partial denture casting alloy. Current methods for bonding acrylic resin to metal alloy can be categorized as mechanical, chemical or a combination of both. Macro-mechanical retentive features using beads, post, open lattice or mesh reduce the bulk of acrylic resin thus compromising retention and esthetics. Micro-mechanical retention can be achieved with acid and electrolytic etching and sandblasting. Chemical bonding is considered necessary to overcome interface denture failures especially in situations such as limited interridge space, short span area and where excessive functional forces are anticipated [9]. Chemical method of bonding has been widely accepted for resin-metal interface due to their ease of manipulation over mechanical methods. Many authors have suggested that use of primers as a means of chemical bonding enhances the bond strength of metal resin interface [6,10,11].

A primer that employs a sulfur monomer together with a phosphate monomer is designated as dual-function primer. The dual-function primer is effective in bonding with both noble and base metal alloys [12]. A dual-function primer (Alloy Primer, Kuraray Medical Co. Ltd., Tokyo, Japan) that contains VBATDT and 10-methacryloxydecyl dihydrogen phosphate (MDP) was used in this study to enhance the adhesion between alloy and heat cure acrylic resin. The primer is a vinylthione coupling agent having principle ingredients as acetone, MDP [10-methacrylicdecyl dihydrogen phosphate],

VBATDT [6-4-vinylbenzene-*n*-propyl-1,3,5-triazine-4-dithione] and a thione-thiol tautomer.

The coupling mechanism of this functional monomer comprises of two stages:

- (i) Transformation of thione ($-C=S$) to thiol ($-C-S-H$) groups on metal surface (M) and subsequently primary bond formation ($-C-S-M$) and
- (ii) Co-polymerization of vinyl groups with the methacrylate-based resin monomer [13].

The MDP monomer consists of three differently functioning components; methacryloyl, decyl and dihydrogen phosphate groups. Of these, the methacryloyl group is undoubtedly indispensable to copolymerize the MDP monomers in the primer and the matrix monomers in the opaque material. Taire et al [14] stated that the principal constituent MDP reacts with the oxide film produced on the metal surface which contributes to increased bond strength and durability. Sulfur atom of VBATDT bonds chemically to precious metal atoms while the double bonds on the other end of the molecule copolymerize with the resin monomers. The presence of MDP promotes the reaction between VBATDT and precious metals resulting in increased bond strength. The

thione tautomer increases the shelf-life of the coupling agent and minimized thiol interferences with resin polymerization. Yoshida et al stated that MDP and VBATDT, having a hydrogen phosphate group, yielded higher bond strength [13].

Comparison of sandblasted Ti and sandblasted Co-Cr

Air abrasion can create suitable surface roughness and increase the wettability of the metal surface thus increasing the mechanical bond strength between the metal and the acrylic resin. Various particle sizes of alumina such as 50 μ m [NaBadalung1998], 110 μ m [Podder 2014], 250 μ m [Kim 2009] have been used for air abrasion. 110 μ m alumina is commonly used for partial denture fabrication. Lim H P [22], Kim S S [18] have found 250 μ m alumina produced optimal surface roughness. Hence 250 μ m alumina particles were used in this study. In this present in-vitro study, the shear bond strength (SBS) between Sandblasted Titanium- Group I (Mean = 2.04 MPa) and Sandblasted Cobalt-Chromium - Group III (Mean = 1.93 MPa) showed no statistically significant difference ($p=0.331$).

A. Comparison of sandblasted and primed Ti with sandblasted and primed Co-Cr

It was observed that chemical treatment after sandblasting resulted in higher shear bond strength values than sandblasting alone. This may be attributed to the activation of the surface energy by roughening of the metal surface. In this in-vitro study, the shear bond strength between Sandblasted and Primed Titanium- Group II (Mean = 2.72 MPa) and Sandblasted and Primed Cobalt-Chromium- Group IV (Mean = 2.49 MPa) showed statistically significant difference ($p=0.001$). The reason for higher bond strength obtained with Titanium may be due to the fact that dihydrogen phosphate group chemically bonds to titanium oxide on the titanium metal, probably bonding covalently or ionically by condensing dihydrogen oxide. The MDP monomer thus appears to be far more effective in conditioning Titanium than cobalt chromium alloy [15]. Lim H P has stated that the VBATDT containing metal conditioner had a significantly positive effect on the bond between heat cure denture base resin and Titanium [6]. Oxidation of alloys is an important aid in obtaining a good bond between denture base resin and alloy. Titanium has the ability to form a stable oxide layer of 10 \AA thickness instantaneously, which may be the reason for increased SBS values obtained in the study [16].

B. Intra-group analysis

When comparing SBS values within Ti-Group I & II and within Co-Cr-Group III & IV, it was found that sandblasted and primed samples in both the groups showed significantly higher values than sandblasting alone. This could be due to the fact that the bonding generated by alumina air abrasion is mechanical whereas the bonding

generated by the alloy primer is chemico-mechanical. Studies have confirmed that chemico-mechanical bonding is superior than mechanical bonding alone. The results are in accordance with the earlier studies by Lim H P [6]

C. Mode of Bond failure

The present in-vitro study, also compared the type of bond failure using stereo zoom macroscope X10 magnification. Adhesive type of bond failure was noted with all the samples of sand blasted Titanium (Group I) and Cobalt-Chromium (Group III).

Mixed type of failure alone was noted with all the samples of sandblasted and Primed Titanium (Group II). Wherein, more of mixed type (90%) and less adhesive type (10%) was found with sandblasted and primed Cobalt-Chromium (Group IV).

The results of the study showed that sandblasting along with the use of primer had better shear bond strength than sandblasting alone for Titanium and Cobalt-Chromium. However, Titanium showed significantly higher shear bond strength values than Cobalt-Chromium when chemico-mechanical treatment was done which could be attributed to the greater wettability of Titanium by Alloy

Primer and the stable oxide film formed. Sandblasting results in removal of the debris as well as metal oxide layer, however due to the reactivity of titanium, even though the oxide layer is removed from the surface, immediate reoxidation of titanium takes place. Hence Titanium samples had better bonding to heat cured denture base resin when compared to Cobalt chromium samples.

Coming to the short comings of the study, factors existing in oral environment such as dynamic fatigue loading, metal framework design, pH changes and the thickness of metal oxide layer, the primer thickness and the difference in depth of roughness created by sandblasting, and after primer application were not considered in this study.

CONCLUSION

Within the limitations of this study, the shear bond strength of PMMA heat cured denture base resin to cast titanium and cobalt-chromium alloy can be improved by the application of metal primers along with air abrasion. However, this is an in-vitro study, the pitfalls may be included in further studies for wide use of clinical application.

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