

## Fatigue Properties of Soldered Minalux and Verabond II Ni Base Dental Alloys

<sup>1</sup>S. Mir Mohammad Rezaei, <sup>1</sup>F. Gramipanah, <sup>2</sup>A. Fallahi Arezodar and <sup>1</sup>A. Amini

<sup>1</sup>Dental School, Tehran University of Medical Science, Tehran, Iran

<sup>2</sup>Department of Mechanic, Amirkabir University of Technology, Tehran, Iran

**Abstract:** Fatigue failure of base metal solder joints are an unsolved problem and information relating to the fatigue strength of solder joints is rarely provided. The purpose of this report is to test the application of Vera solder for use with the base metals of Verabond II and Minalux and its effect on fatigue behavior of these joints. Fifteen cast and 12 soldered joint specimens were made of each alloy. The thermal cycles required for oxidation, opaque, dentin and glazing were performed. Rotating, bending and cantilever fatigue tests were performed with a 2880 rpm (ASTM Rotating bending fatigue testing of metals JIsz 22 74;1078). All soldered specimens except two in each group fractured at soldered joints. All of the specimens from intact Verabond II and Minalux fractured at the gripping point of machine except two Minalux specimens that fractured at the midpoint. Under equal load, the number of loading cycles to cause fracture in intact Verabond II is greater than in intact Minalux. In soldered Verabond II the fatigue limit was 20 MPa higher than in soldered Minalux. With probability of 90% and with Confidence Interval (CI) of 90% the fatigue limit of soldered Minalux is 194.65MPa and for soldered Verabond 2, it is not possible to give a probability. The Fatigue Ratio of soldered Verabond 2 is 0.57 and Minalux 0.50. Due to the influences of several parameters, with use of the gas-oxygen torch, the resulting solder especially in base metal alloy is not reliable. As a result, Vera solder alloy by gas-oxygen torch is not recommended for Verabond II and Minalux.

**Key words:** Rotating bending, solder, Ni Cr alloys, mechanical properties

### INTRODUCTION

Earlier reports on the tensile strength of base metal solder joints used for large and small dental castings indicate that they are stronger than precious metal solder joints (MacEntee *et al.*, 1981; Hawbolt *et al.*, 1983). The clinical relevance of this information must be questioned when the brittle characteristics of base metal alloys are considered. The result of a static load test may not be a reliable predictor for clinical behavior under the fatigue conditions produced in the mouth (Hawbolt and MacEntee, 1983). Little is known of the mechanical resistance of solder joints under cyclic loading. Contrary to the results of monotonic tests, fatigue data are generated by application of comparatively small cyclic stress to a specimen. Such a test is commonly reported by plotting the applied stress levels on the Y- axis and the number of cycles until failure on the abscissa (S-N diagram). For the most materials, the number of cycles to failure will be fairly high when the applied stresses are small, while the specimen will fail almost immediately when the stress level lies in the vicinity of the ultimate tensile strength (Arezodar, 1985). Some materials present a peculiar behavior such that they withstand an almost

infinite number of cycles when the applied stress is kept below a certain level (endurance limit). In these instances, the endurance limit is determined by the asymptotic line to which the S-N curve flattens out (Wiskott *et al.*, 1991). In the fixed partial denture, soldered joints are the weakest parts of framework. Thus, the present research reports an investigation of the fatigue properties of soldered base metal alloys.

### MATERIALS AND METHODS

The hypothesis in this study is to examine if Vera solder which is recommended for the the verabond II could be used for Minalux or not, The effect of this solder on the fatigue properties of these two type of alloys were also investigated.

The compositions of dental Ni-Cr alloys and dental solders used in this study are shown in Table 1 and 2. For tensile test, 4 specimens were made for each of two intact groups for the total of 8 specimens were examined. One specimen in each group received a thermal history to simulate porcelain application. One specimen from the soldered groups was examined by a tensile test. They received thermal history to simulate porcelain application.

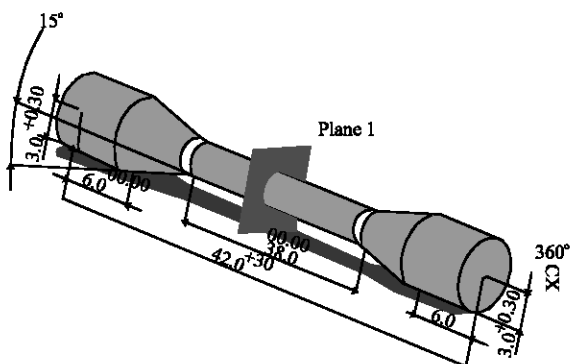


Fig. 1: Fatigue specimen geometry showing section of soldering plane (plane 1)

Table 1: The composition of minalux and verabond II.

Alloys	Ni (%)	Cr (%)	Mo (%)	Al (%)	Ti (%)	Nb (%)	Si (%)
Verabond 2	77.5	12.5	4.25	2.25	0.45	4	0.5
Minalux	73	13	4	2	0.5	4.5	3

Table 2: The composition of vera solder

B (%)	Fe (%)	Si (%)	Cr (%)	Ni (%)
3	4	4.5	12	75

Two soldered groups and 2 intact groups of Verabond 2 and Minalux 6 specimens for each of 4 groups for a total of 24 specimens were examined for tensile test in a previous reaserch (Mir Mohammad Rezaee STNA, 2002). The specimens were tested for UTS using an universal Instron testing machine with the crosshead speed set at  $0.5 \text{ mm min}^{-1}$ .

Fifteen cast and 12 solder joint specimens were made of each alloy. For specimen preparation, wax patterns with a radius of  $3.0 \pm 0.1 \text{ mm}$ , 15 mm length and 6 mm minimum end conical section were prepared (Fig. 1) by injection of blue inlay casting wax (Kerr Peterborough, UK) into a split brass mold as recommended by ANSI/ADA specification. These wax patterns were invested in a phosphate bonded investment (Degovest HFG, Deyusa Co. Germany) and cast with a gas oxygen multiorifice torch in Minalux (Mavad Karan Co.- Iran) and Vera bond 2 (Alba Dent Co. USA). After casting, the rings were bench cooled and the castings divested and cleaned with  $50 \mu\text{m SiO}_2$  at 25 PSI. Any nodules were carefully removed with stone and carborundum disk and polished with 600, 800 and 1000 grit silicon carbide sand paper (Carbimat paper, Buehler Ltd). Each specimen was radiographed to determine internal porosity. Defective castings were discarded and new specimens were made. A custom made cutting jig allowed each casting to be cut in half. A positioning jig ensured that the long axis of casting was mounted perpendicular to the cutting edge of the disk and the soldering gap adjusted to 0.3 mm. The gap was filled with

autopolymerizing resin (Duralay, Reliance Mfg, Worth III and Illinois) and allowed to bench set for 30 min. The rod resin assembly was invested for soldering (Deguvest HFG). After one hour, the resin was burned out at  $250^\circ\text{C}$ . After cooling, the gap was cleaned with a sable brush and tap water and the investment was allowed to dry. For soldering, the gap was fluxed (Vera Flux, Albadent USA) and inserted in the front of a furnace adjusted to  $150^\circ\text{C}$  for 10 min. Then the block was heated at  $930^\circ\text{C}$  for 15 min and brought to a bright red color with a gas-oxygen torch. The solder was allowed to flow into the joint space (Vera solder, Albadent, USA). The specimen was bench cooled, divested and sand blasted with  $\text{Al}_2\text{O}_3$  at 90 pounds pressure. Extra solder was removed using a handpiece and green abrasive stone at low speed. The solder joint area was refined with 320, 500 and 1000 grit silicon paper. The diameter of each rod was recorded as the average of 3 measurements made for each specimen using a micrometer accurate to 0.01 mm. For both the soldered and the cast specimens a radiographic examination was performed. Specimens were exposed to an x-ray beam with an accelerating voltage of 90 KV and a current of 10 mA for 1 sec. An additional film was exposed after rotating the specimen at a  $90^\circ$  angle to the initial orientation (DeHoff *et al.*, 1990). Then the specimens were inspected at 20X magnification. All specimens with surface defects were eliminated. The thermal cycles required for oxidation, opaque, dentine and glaze were performed as follows: oxidation ( $650\text{-}960^\circ\text{C}$ ), opaque ( $540^\circ\text{-}960^\circ\text{C}$ ), dentin ( $540^\circ\text{-}940^\circ\text{C}$ ) and glazing ( $540^\circ\text{-}920^\circ\text{C}$ ) (DeHoff *et al.*, 1990).

## RESULTS

The UTS values for each group of specimens and tensile test of previous work are shown in Table 3.

In the fatigue test, if the specimen was not fractured under 2, 500, 000 cycles loading, it achieves the fatigue limit. In the intact Verabond group, 9 specimens were tested and 9 points of SN curve were determined, The results are shown in Table 4 and Fig. 2, where  $\sigma$  is applied stress, N the number of cycles. The fatigue limit was measured at 370 MPa for the intact Verabond II. Ten specimens of intact Minalux were tested and the results are shown in Table 5 and the S-N curve determined for intact Minalux are determined, in Fig. 3. Specimens number 6, 8 and 4 fractured under low load because of defects. The fatigue limit for this group was 350 MPa under 3, 100, 000 cycles. The results for 10specimens of soldered Verabond II are shown in Table 6. The fatigue limit for this group was 250 MPa under 2, 560, 000 cycles.

Table 3: UTS. value of different specimen groups

Group	Ys	UTS MPa	UTS previous Mpa
Int. V	329.88	715.68	686±116
Int. M	309.24	624.63	732±128
Int. V. H		690.93	
Int. M. H	384.26	709.45	
S. V. H	256.16	492.11	
S. M. H		402.54	
S.V			432±51
S.M			308±173

M: Minalux, V: Verabond II In: Intact, H: Heat treatment, S: soldered

Table 4: Fatigue test result in intact verabond II

Result	N	σ MPa	Specimen number
Fractured specimen	54973	520	1
Fractured specimen	1100000	380	2
Fractured specimen	71694	570	3
Fatigue limit			
Did not fractured	3150000	370	4
Fractured specimen	30708	610	5
Fractured specimen	369367	470	6
Fractured specimen	123000	420	7
Fractured specimen	213794	400	8
Fractured specimen	412245	400	9

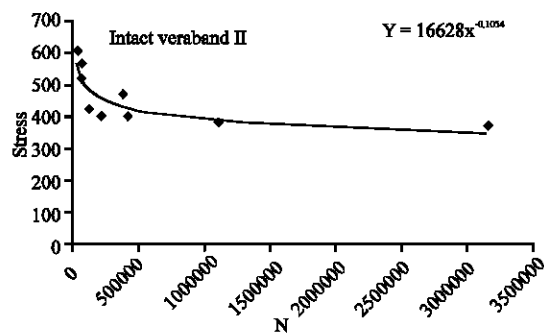


Fig. 2: S-N curve of intact Verabond II (stress y MPa and N number of cycle to failure)

The result of 10 specimens of soldered Minalux is shown in Table 7. Four specimens of this group were fractured at the starting point of the test. The fatigue limit of this group was 230 MPa under 25, 600, 000 cycles. Other results include:

- All intact Verabond II specimens fractured at the gripping point of the machine and in the greatest diameter of the cross section.
- All intact Minalux specimens fractured at the gripping point of the specimen except 2 specimens that fractured at the midpoint.
- The Standard Deviation (SD for intact Minalux is greater than for intact Verabond II.
- Under equal load, the number of loading cycles to cause fracture in intact Verabond II was greater than in intact Minalux. The fatigue limit for intact Minalux is 20 MPa less than Verabond II.

Table 5: Fatigue test result in intact minalux

Result	N	σ MPa	Specimen number
Fatigue limit, not fractured	3100000	350	1
Fractured specimen	147150	400	2
Fractured specimen	615000	385	3
Fractured specimen	105000	360	4
Fracture specimen	87869	420	5
Fractured specimen	2800	380	6
Fractured specimen	1390585	370	7
Fractured specimen	5230	360	8
Fractured specimen	983	570	9
Fractured specimen	62855	520	10

Table 6: Fatigue test result in soldered verabond II

Result	N	σ MPa	Specimen number
Fractured specimen	300	270	1
Fractured specimen	458	400	2
Fractured specimen	410	250	3
Not fractured fatigue limit	2500000	250	4
Fracture specimen	170	310	5
Fractured specimen	277	350	6
Fractured specimen	315	270	7
Fractured specimen	253	250	8
Fractured specimen	100	250	9
Fractured specimen	130	250	10

Table 7: The result of fatigue test in soldered minalux

Result	N	σ MPa	Specimen number
Fatigue limit	135	275	1
Fractured specimen	1665250	250	2
Fractured specimen	100	275	3
Fractured specimen	215	250	4
Fracture specimen	2000800	230	5
Fractured specimen	120	240	6
Fractured specimen	1364000	260	7
Fractured specimen	310	270	8
Not fractured fatigue limit	2560000	230	9
Fractured specimen	434	240	10

N = Number of cycle, σ = stress

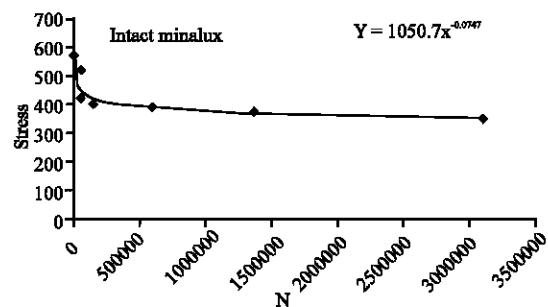


Fig. 3: S-N curve of intact Minalux (stress y MPa and N number of cycles to failure)

- All of the soldered Verabond II specimens fractured at the joint except one specimen which at a point far from the joint (no. 6) and one specimen (No. 4) which reached endurance fatigue limit and did not fractured.
- All Minalux soldered joints fractured at the joint except one (no. 2) which at a point far from the joint and one specimen (no.9) which reached the fatigue limit and did not fracture.

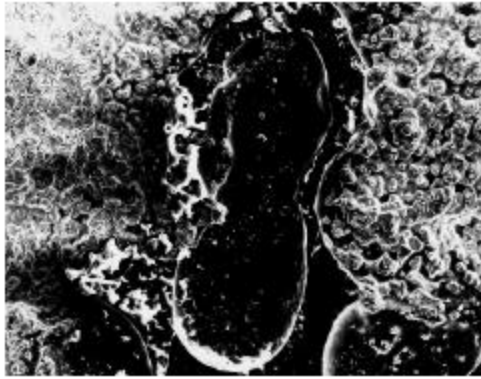


Fig. 4: SEM soldered Verabond II, porosity and microcrack in the center of specimen ( $\sigma = 250$  MPa,  $N = 410$ ), X 100



Fig. 5: SEM fracture surface of soldered Minalux specimen no. 2 ( $N=1665250$  cycle,  $\sigma = 250$  MPa), X 125

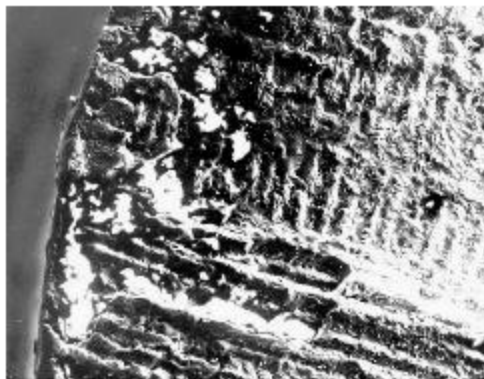


Fig. 6: Flux observed in the SEM of soldered Minalux specimen no. 6 leading to low cycle fracture ( $N = 120$  cycle  $\sigma = 240$  MPa), X 100

- In 5 specimens of soldered Verabond II, those tested under 250 MPa load, fracture probability was found to be 80%. The inclusion of voids and porosity as

Table 8: The result of fatigue ratio in 4 groups

Groups	Fatigue limit MPa	UTS MPa	Fatigue Ratio $S/S_u$
Intact verabond II	370	690.93	0.53
Intact minalux	350	709.45	0.44
Soldered verabond II	250	492.11	0.50
Soldered minalux	230	402.54	0.57

shown in Fig. 4 resulted in the low cycle fracture of this load. So under ideal conditions for the torch soldering method, 250 MPa is the fatigue limit.

- Two soldered Minalux specimens were tested under 230 MPa load and survived beyond 2, 000, 000 cycles, no specimen fractured so 230 MPa is the fatigue limit, 20 MPa less than Verabond II. Fracture probability for this alloy is less than for Verabond II.
- Of 3 specimens of soldered Minalux tested in the range of 250-260 MPa, 2 specimens survived more than 1, 364, 000 cycles. This suggests that in this range long life probability for soldered Minalux is 75 % (Fig. 5). In addition, the two specimens of No 6 and 10 tested at 240 MPa exhibited low cycle fracture. The origin of crack initiation from flux observed in the fracture surface shown in the Fig. 6, it seems to be the reason for this low cycle fracture.

Data analysis on Minalux soldered group were performed according to the guideline for fatigue testing and statistical analysis. Four specimens were survived more than  $10^6$  cycles (Ralph, 2001). The Mean value and Standard Deviation (SD) was calculated.

$$\bar{x} = 242.5, SD = 5$$

According to this guideline with assuming normal distribution and with probability of %90 and with Confidence Interval (CI) of %90 ( $n = 4$ )  $k$  value is 3.19. The predicted fatigue limit is then calculated from Eq. 1:

$$\begin{aligned} x &= \bar{x} - k(SD) \\ x &= 242.5 - 3.19(5) \\ x &= 194.95 \end{aligned}$$

In the soldered Verabond II group one sample was survived more than  $10^6$  cycles, so it is not possible to give a probability. SN curve of intact Verabond II (Fig. 2).  $S = 1662N^{-0.166}$  exhibits a higher fatigue strength coefficient of 1662 than for the intact Minalux of 1050. Fatigue strength exponent is measured for the Verabond II  $b = -0.105$  compared with the value of  $b = -0.075$  for the Minalux, where  $b$  was reported for SAE 4340 steel  $b = -0.09$  (Hertsberg 1983).

Fatigue Ratio is the ratio of fatigue strength or fatigue limit ( $S_f$ ) to Tensile Strength ( $S_u$ ). For many materials, Fatigue ratio may be used to estimate fatigue properties from data obtained in tension tests. Considering fatigue limit based on a normal alternating stress, this value has ranged 1-70% of the ultimate tensile strength. The  $S_f/S_u$  ratio is called "fatigue Ratio". The result of Fatigue Ratio is shown in Table 8.

## DISCUSSION

Ni Cr alloys has much higher value of modulus of elasticity compared to the noble metal alloys. Therefore long span fixed prostheses fabricated from Ni-Cr alloys will undergo much less flexure than similar prostheses fabricated from noble metal alloys, with less likelihood of fracture of the brittle dental porcelain component. These base metal casting alloys are generally considered more technique-sensitive and difficult to cast than the noble metal casting alloys (Rossenstiel, 2001). A basic method of presenting engineering fatigue data is in terms of an S-N curve which represents the dependence of the life of the specimen on the number of cycles to failure (N) at the maximum applied stress. First, the cyclic life of an axle increases with a decreasing stress level and below a certain stress level, the life appears to be infinite; second, the fatigue life is reduced drastically by the presence of a notch (Scherrer *et al.*, 2003). Rotating, Bending, Cantilever Fatigue tests (RBCF) rest on the premise that the occlusal surface of natural teeth or dental prosthesis is subjected to a field of force vector that spans 180°. Teeth or restorations may be loaded with forces that are active buccolingually or occlusoapically or a combination of these two. In essence, RBCF tests add the remaining 180° to complete a 360° circle. This method of stress calibration is described in detail by Amini (Amini, 2002).

In this area, two main avenues of research have been followed. First, several reports have examined the structure of the interfaces between the solder and parent metals through microprobe analysis or optical microscopy or electron microscopy of metallography specimens (Walters, 1976). In the second avenue of investigation, different parent metal solder combinations have been subjected to monotonic force application and the resultant fracture strength recorded (Janus *et al.*, 1983). Yet, although they are quite pertinent, fatigue tests still present a number of limitations. First, the lack of standardization of the testing protocols and analytical procedures prevents direct inter-study comparisons. Indeed, in addition to rotational fatigue, reversed stressing in 3-point bending. Braem *et al.* (1994a, b, 1995) cyclic loading (on/off type) of 4-point bend bars (Dummond and Miescke, 1991) repetitive impact loading

(Htang *et al.*, 1995) and combinations of monotonic and fatigue tests have been used by different research groups. Second, the distribution of raw fatigue data is non-normal and therefore requires specific analytical procedures. Standardization is also needed. Third, fatigue tests are cumbersome in that larger specimen numbers and significantly more time are required than in monotonic tests (DeHoff *et al.*, 1990). Still, it is the authors' contention that rotational fatigue tests as conducted in this study represent a near-optimum method for the testing of dental materials and structures. Because of the specimen's rotation, such tests are self-optimizing in that they will locate the point of the 360° circumference where stress concentrations are highest and where fracture will begin. Further, rotational bending tests are applicable to a variety of materials and to entire prosthodontic structures as long as the specimens are configured as rotation-symmetric.

One of our goals was the comparison of fatigue resistance of intact Minalux and Verabond II and soldered joint specimens of those materials.

The composition of the parent alloys are shown in Table 1 and the composition of solder alloys in Table 2. Although, in accordance with the standard, the manufacturer should report the main elements of the alloys as well as trace elements, the manufacturer has not revealed the details of composition. The composition of Minalux and Verabond II is the same except that the Si in Minalux is 6 times greater than Verabond II. The result of the UTS tests in some specimens was low because of porosity or voids in soldered joints or in casting or the high temperature of soldering.

In accordance with the ASTM for fatigue tests, the surfaces of specimens should be polished which may cause stress concentration in some specimens and decrease fatigue resistance. In the clinical situation, it is not possible to polish the inner surface of the crown so the fatigue strength must be considered lower than in this study. Luotio and Vallittu (1996) have studied the fatigue test of Ti. They have suggested that fatigue strength is proportional to hardness. All except two of the soldered specimens fractured at the soldered joint demonstrating that solder joints commonly show less resistance.

For all materials, the number of cycles to failure will be fairly high when the applied stresses are small, while the specimens will fail almost immediately when the stress level lies in the vicinity of the ultimate tensile stress. Intact specimens of the alloys fractured early when applied stress was in vicinity of the UTS and also soldered specimens fractured at the stress range of lower than Yield Strength (Y.S) Fatigue failure dependent on a random structural defect pattern inside a polycrystalline, aggregate heterogeneous structure of solder joints may

have increased the scatter even further, suggesting that the solder-alloy combination should be evaluated not only with respect to its monotonic fracture resistance, but equally by considering the shape of the S-N curve alloy. Solder combinations with high UTS but a rapidly falling S-N curve should be rejected.

The results of this study show that Vera solder is not a compatible solder for either alloys with the torch method. Specimen no. 2, soldered Minalux loaded at 250 MPa, surpassed 1,665,250 cycles before breaking (Fig. 5) while specimen no. 6, soldered Minalux, fractured at 240 MPa after 120 cycles because of impurities shown in (Fig. 6) which originated mainly from flux during torch soldering method. It seems likely that particles of flux may be incorporated into the joint during melting of the solder. In the SEM study, those specimens which fractured far from the joints at the low cycle revealed porosity in fractography leading to micro-cracking (Fig. 4). The microscopic imperfections were not detected visually or with radiography. Other factors such as low wetting of parent metals and porosity, excessive temperatures and overheating and an interaction between the alloy and the constituents of the investment will affect torch soldering methods of joining metals (O'Brien, 1969).

Fatigue properties are frequently correlated with tensile properties. In general, the fatigue limit of cast and wrought steels is approximately 50% of the ultimate tensile strength. The ratio of the fatigue limit (or the fatigue strength at  $10^6$  cycles) to the tensile strength is called the fatigue ratio.

Several nonferrous metals such as nickel, copper and magnesium have a fatigue ratio of about 0.35. While the use of correlations of this type is convenient, it should be clearly understood that these constant factors between fatigue limit and tensile strength are only approximations and hold only for the restricted condition of smooth, polished specimens which have been tested under zero mean stress at room temperature. Regardless of stress level, fatigue cracks initiated at the specimen surface in intergranular or transgranular mode due to cyclic slip deformation at a very early stage of fatigue cycling. In these alloys fatigue ratio is between 0.44-0.53 for intact groups and 0.50-0.57 for soldered groups.

In a typical long-span dental bridge, the tensile stress will occur on the gingival surface at the artificial tooth-solder interface. Chewing forces seldom exceed 6.9 Kg and most forces are less than 1.6 kg. Usually this force is applied periodically and amounts to about 17.5 min of occlusal contact per day with a rate of chewing rarely greater than 120 cycles  $\text{min}^{-1}$ . Therefore, it is reasonable to assume that occlusal forces on a bridge will be applied for 2100 cycles  $\text{day}^{-1}$  with 105 cycle  $\text{day}^{-1}$  at peak force and 1995 cycles  $\text{day}^{-1}$  at 2 kg forces. The tensile stress on the lower beam surfaces (due to loading) has been calculated to equal 240 MPa (Walters, 1976). In this study,

the fatigue limit of soldered Verabond II is measured higher than 240 MPa, so the risk of fracture in Verabond II is low. The fatigue limit in Minalux was 230 MPa. This is very close to 240 MPa, considering that 3 soldered Minalux specimens at 250 MPa and 260 MPa and 230 MPa survived more than 1,300,000 cycles before breaking and the fatigue limit is measured at 230 MPa. So, if the length of a bridge's span shortens, the tensile stress is reduced and fatigue strength may be adequate. Similarly, uniform occlusal contacts along the bridges surface may distribute the load and reduce the stress on the soldered joints. If the abutment teeth are mobile, higher bending stresses would be experienced by the solder joints and the bridges will fail earlier (Graf, 1969).

## CONCLUSION

The fatigue properties of Minalux and Verabond II were tested in this study. The fatigue strength of intact Verabond II is 20 MPa higher than for Minalux. In soldered specimens, the fatigue limit of soldered Verabond II is 20 MPa higher than soldered Minalux. In the gas-oxygen torch method of soldering, the influences of several parameters, such as impurities which originate mainly from flux, may be incorporated into the joint during the melting of the solder. The resulting solder, especially in base metal alloy, is not reliable. For this reason, Vera solder alloy is not recommended for soldering Verabond II and Minalux by the gas-oxygen torch method.

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