Fatigue Properties of Cast Ag–Pd–Cu–Au–Zn Alloy for Dental Applications in the Relation with Casting Defects

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Ag–Pd–Cu–Au–Zn type alloys have been widely used as dental prosthetic materials. In general, the dental prosthetic products are fabricated by a dental casting method. The dental castings contain the casting defects such as micro shrinkages, pores, surface roughness, etc. These defects cause fatigue stress due to the mastication applied to the dental prosthesis in the practical use. Therefore, the effects of the casting defects on the properties of the cast alloy become to be a fatigue crack initiation site as an initial crack. This means that the size of the shrinkage affects the fatigue strength of this cast alloy. The tolerant size of the shrinkage that satisfies the target value of the fatigue limit (230 MPa) of this cast alloy is calculated by using the equation derived in this study, which describes the relationship between the maximum stress intensity factor and the number of cycles to failure.

1. Introduction

Dental Ag–Pd–Cu–Au–Zn type alloys have been widely used as dental prosthetic materials in Japan. However, since restorations made by these type alloys are often fractured by cyclic mastication, that is, fatigue. The example of the fatigue fracture of the dental alloy is shown in Table 1. Therefore, it is necessary to know mechanical properties, in particular, the fatigue properties of dental alloys. Fundamental studies of the fatigue properties in the relation with microstructure have so far been done about a dental Ag–Pd–Cu–Au–Zn type alloy also actually exists. Therefore, it is necessary to know mechanical properties, in particular, the fatigue properties of dental alloys. Fundamental studies of the fatigue properties of the cast alloy are investigated in this study, which describes the relationship between the maximum stress intensity factor and the number of cycles to failure.

2. Experimental Procedures

2.1 Material

The materials used in this study were commercial dental Ag–Pd–Cu–Au–Zn type alloys. The chemical composition of this alloy is shown in Table 1.

Table 1 Chemical composition of Ag–Pd–Cu–Au–Zn alloy.

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<tr>
<th></th>
<th>Ag</th>
<th>Pd</th>
<th>Cu</th>
<th>Au</th>
<th>Zn</th>
<th>Other</th>
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<td></td>
<td>mass%</td>
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<tr>
<td>Ag</td>
<td>51.0</td>
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<tr>
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<tr>
<td>Other</td>
<td>0.5</td>
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The drawn bars were cast into tensile and fatigue specimens by a lost wax method as will be mentioned below.

Some of the drawn alloy bars were solutionized at 1073 K for 3.6 ks in vacuum, and then cooled in air. This heat treatment was found to lead to good balance of tensile properties, fracture toughness and fatigue properties in the previous study. This heat treatment condition may give a similar matrix microstructure to that of casting alloy. From these heat-treated bars, tensile and fatigue specimens were machined for a comparison with casting ones as will be also mentioned below.

2.2 Casting of specimen
Casting of tensile or fatigue specimen was carried out using a lost-wax method. The acrylics stick with a diameter of 6 mm and a length of 50 mm was machined into the geometry of the tensile specimen with a gauge diameter of 3 mm and a gauge length of 20 mm or glass type fatigue specimen with a minimum diameter of 3 mm, and it was used as a wax pattern (casting prototype). The investment material used in this study was a plaster type one. The casting mold was left in air for 24 hours after investing. Then, the mold was heated to 973 K at a heating rate of 5 K/min for firing. Casting was carried out using a high frequency vacuum pressure-casting machine, and then casting specimen with mold was cooled in air at room temperature (295 K). The specimen surfaces were sand blasted.

The surface of some fatigue test specimens were finished by buff-polishing in order to investigate the effect of surface roughness on the fatigue properties. The as-blasted and buff polished cast specimens will be designated as “Non-polished cast specimen” and “Polished cast specimen” hereafter.

2.3 Specimen for drawn alloy bar
Tensile specimens with a gauge diameter of 2 mm and a gauge length of 20 mm, and fatigue specimens with a gauge diameter of 5 mm and a gauge length of 20 mm were machined from the heat-treated drawn alloy bars mentioned above. In this case, the surface of the gauge area of the tensile specimen was polished using a water-proof emery paper up to #1500, while the specimen surface of the fatigue specimen was finished by buff polishing. The specimen machined from the heat-treated drawn bar will be designated as “D-1073AC drawn specimen” hereafter.

2.4 Observation of microstructure
The microstructure of each specimen was characterized by the backscatter electron image (BSE). The volume fractions of constitutional phases, that is, $\alpha_1$, $\alpha_2$ and $\beta$ phases were measured on the BSE image micrographs using a pattern analysis software.

2.5 Measurement of surface roughness
The surface roughness of the Non-polished and Polished cast fatigue specimens were measured using a surface character analyzer.

2.6 Tensile and fatigue tests
Tensile tests were carried out using an Instron type machine at a crosshead speed of $8.33 \times 10^{-6}$ m/s in air at room temperature (295 K). The load was measured by a load cell located in the testing machine. The displacement was measured using a strain gauge directly attached to the parallel part of the specimen and a reading microscope.

Fatigue tests were carried out in order to produce a S–N curve for each specimen at a stress ratio, $R$, of 0.1 and a frequency of 10 Hz with a sine wave in air at room temperature (295 K).

2.7 Observation of fracture surface
Observation of fracture surface after tensile or fatigue test was carried out using a scanning electron microscope (SEM). In this case, the specimens were cut at a distance of about 2 mm from the fracture surface. Each fracture surface was subjected to SEM observation after ultrasonic cleaning in acetone.

2.8 Measurement of ratio of casting defect
Casting defects, pores and micro shrinkages, were observed on the fracture surface using an SEM, and the ratio of each casting defect was measured on the fractograph. In this case, the diameter of casting defect over 10 $\mu$m was measured because the casting defect smaller than 10 $\mu$m was difficult to distinguish from dimples. The casting defects were also observed using an optical microscope on the polished sample of the cross section near the fracture surface, and their ratios were measured on the micrograph.

2.9 Calculation of stress intensity factor
The casting defect identified as a crack initiation site by the fatigue fracture surface observation was approximated as an ellipse based on the route area method as shown in Fig. 1. The approximated ellipse was assumed as an initial crack, and then the maximum cyclic stress intensity factor, $K_{I_{\text{max}}}$, was calculated using the following eq. (1).

$$K_{I_{\text{max}}} \approx 0.65 \times \sigma_{\text{max}} \sqrt{\pi C D A}$$

where $\pi$, $\sigma_{\text{max}}$, and $C D A$ are the ratio of the circumference of a circle to its diameter, the maximum cyclic stress and the approximated ellipse area, respectively.

![Fig. 1 Schematic drawings of (a) maximum stress intensity factor for casting defect and (b) area approximation of casting defect.](Image)
3. Results and Discussion

3.1 Microstructure

BSE photographs of the microstructure of the central part of the cast specimen and the microstructure near the surface of the cast specimen and D-1073AC drawn specimen were shown in Figs. 2(a)–(c), respectively. The microstructure of dental Ag–Pd–Cu–Au–Zn alloy is, in general, composed of three phases. They are \( \alpha_1 \) phase that is copper-rich matrix, \( \alpha_2 \) phase that is silver-rich matrix, and Cu–Pd intermetallic compound, that is, \( \beta \) phase that precipitates during relatively slower cooling from solutionizing temperature or aging treatment. In this figure, the white portion distributed over each microstructure is Ag-rich \( \alpha_2 \) phase that is one of the matrix phases, and the black portion is Cu-rich \( \alpha_1 \) phase that is another matrix phase. It is known that both phases have fcc structures.\(^{10}\) Moreover fine black precipitates with a diameter of around 1 \( \mu m \) are distributed in each microstructure. These are \( \beta \) phases, which have been reported to be PdCu\(_X\)Zn\(_X\) system intermetallic compounds.\(^{11}\)

Comparing the microstructure of cast specimen (Fig. 2(a) and Fig. 2(b)) with that of D-1073AC drawn specimen (Fig. 2(c)), \( \alpha_1 \) and \( \beta \) phases in cast specimen are distributed uniformly, but \( \alpha_1 \) and \( \beta \) phases in D-1073AC drawn specimen are distributed uniformly.

Comparing the microstructure of the central part (Fig. 2(a)) of cast specimen with the microstructure near the surface of the cast specimen (Fig. 2(b)), the amount of the black portion, that is, \( \alpha_1 \) phase, is greater in the central part of the specimen than near the specimen surface because the cooling rate of the central part of the specimen is smaller than near the specimen surface.

Comparing the microstructure of cast specimen with that of D-1073AC drawn specimen (Fig. 2(c)), the amount of \( \alpha_1 \) phase is greater and the size of \( \alpha_1 \) phase is also greater in the cast specimen than in D-1073 AC drawn specimen. This is also due to the cooling rate difference between two materials although the effect of drawing exists in the D-1073AC drawn specimen. D-1073AC drawn specimen was cooled with directly touching to air during air cooling after solutionizing, while cast specimen was air cooled in the mold after solutionizing. Therefore, the cooling rate is relatively smaller in the cast specimen than in D-1073AC drawn specimen.

Comparing the microstructures of the central part and near the surface of the cast specimen with the microstructure of the D-1073AC drawn specimen, the amount of \( \beta \) phase precipitated is greater in the cast specimen than in D-1073AC drawn specimen. In the case of the cast specimen, the amount of \( \beta \) phase is greater in the central part than near the specimen surface also because the cooling rate is smaller in the central part than near the specimen surface.

The volume fractions of \( \alpha_1, \alpha_2 \) and \( \beta \) phases are shown in Fig. 3 where the total amount of \( \alpha_1, \alpha_2 \) and \( \beta \) phases is 100%. The volume fractions of \( \alpha_1 \) and \( \beta \) phases are apparently greater in the cast specimen than in D-1073AC drawn specimen.

3.2 Tensile properties of cast specimen

Tensile test results of cast specimen and D-1073AC drawn specimen are shown in Fig. 4. The tensile strength, \( \sigma_B \), and 0.2% proof stress, \( \sigma_{0.2} \), of the cast specimen are greater than those of D-1073AC drawn specimen. However, the elongation of cast specimen is smaller as compared with that of D-1073AC drawn specimen. The scattering of the elongation of the cast specimen is greater as compared with that of D-1073AC drawn specimen. The amounts of intermetallic compound \( \beta \) phase that leads to high strength and low ductility, and Cu rich \( \alpha_1 \) matrix phase that leads to low strength and high ductility are greater in the cast specimen than in D-1073AC drawn specimen. The microstructure of the cast specimen may be coarser than that of D-1073AC drawn specimen. In this study, the effect of precipitation strengthening is predominantly because the strength is greater and the elongation is smaller in the cast specimen than in D-1073AC drawn specimen in this study.

The relationship between the volume fraction and the number of the micro shrinkage measured on the fracture surface
of cast specimen and the elongation is shown in Fig. 5. The correlation between the volume fraction of the micro shrinkage and elongation is not recognized. On the other hand, the correlation between the number of the micro shrinkage and the elongation can be recognized. That is, the elongation decreases with increasing the number of the micro shrinkage.

3.3 Fatigue properties of cast specimen

S–N curves of Non-polished cast specimen, Polished cast specimen and D-1073AC drawn specimen are shown in Fig. 6. The fatigue strength of Non-polished specimen is similar to that of Polished specimen in both low cycle fatigue life region and high cycle fatigue life region. The scatter of the fatigue strength of both cast specimens is greater than that of the fatigue strength of D-1073AC drawn specimen. The fatigue strength of Non-polished cast specimen are 352–492 ($\Delta\sigma_{\text{max}} = 140$) MPa at $10^5$ cycles, while 209–284 ($\Delta\sigma_{\text{max}} = 75$) MPa at $10^6$ cycles. The fatigue strength of Polished cast specimen are 347–565 ($\Delta\sigma_{\text{max}} = 218$) MPa at $10^5$ cycles, while 233–251 ($\Delta\sigma_{\text{max}} = 18$) MPa at $10^6$ cycles. The scatter of the fatigue strength of the cast specimen is considerably large especially in the low cycle fatigue life region where $N_f$ is up to $10^5$ cycles. The fatigue limits, which are the fatigue strength where the number of cycles to failure is over $10^7$ cycles are round 210 MPa. On the other hand, although the fatigue limit of D-1073AC drawn specimen is not obtained, it is expected near 400 MPa.

SEM fractographs near the fatigue crack initiation site of Non-polished and Polished cast specimens and D-1073AC drawn specimen, which have been broken in the high cycle fatigue life region are shown Fig. 7. In the case of D-1073AC drawn specimen, the fatigue crack initiates at the slip band on the specimen surface, but in the case of Non-polished and Polished cast specimens, the fatigue crack initiates from the micro shrinkage near the specimen surface. In general, since the stress constraint is smaller on the specimen surface than in the specimen, slip damage accumulates on the specimen surface, and then extrusion and intrusion occurs there. Then, the stress concentration occurs, and the fatigue crack initiates along the slip plane. Therefore, if polishing scars, defects, etc. exist on the specimen surface, the stress concentration occurs there, and the fatigue strength is lowered. Therefore, it is generally said that the fatigue strength depends strongly on the surface roughness of the specimen.\(^{12}\)

There existed a large difference in the specimen surface roughness between Non-polished cast specimen and Polished cast specimen as shown in Fig. 8. Since the specimen surface roughness of Non-polished cast specimen was greater as compared with that of Polished cast specimen, the fatigue strength of Non-polished cast specimen was expected lower than that of Polished cast specimen. However, as already stated, the fatigue crack initiation site of Non-polished or Polished cast specimen is the micro shrinkage near the specimen surface. Therefore, the stress concentration is more easily occur at the micro shrinkage near the specimen surface than at the specimen surface. In this study, the fatigue strength of the cast specimen does not depend on the specimen surface roughness, but depend on the micro shrinkage near the specimen surface.

The relationships between the fatigue strength of both cast specimen and the volume fraction of the micro shrinkage measured on the fatigue fracture surface are shown in Fig. 9.
\[
\sigma_{\text{max}} = 250\text{MPa} \\
N_f = 9.50 \times 10^5
\]
\[
\sigma_{\text{max}} = 233\text{MPa} \\
N_f = 1.48 \times 10^6
\]
\[
\sigma_{\text{max}} = 496\text{MPa} \\
N_f = 2.89 \times 10^6
\]

Fig. 7 SEM fractographs in the high cycle fatigue life region; (a) Non-polished cast specimen, (b) Polished cast specimen and (c) D-1073AC drawn specimen. Arrows indicate crack initiation sites. \(\sigma_{\text{max}}\) and \(N_f\) are the maximum cyclic stress and the number of cycles to failure.

![Fig. 8 Surface roughness of Non-polished cast specimen and Polished cast specimen.](image)

![Fig. 9 Relationship between fatigue strength and each volume fraction of shrinkage measured on fractograph of cast specimen.](image)

Although the fatigue strength decreases with increasing the volume fraction of the micro shrinkage in the low cycle fatigue life region, the fatigue strength hardly changes with increasing the volume fraction of the micro shrinkage in the high cycle fatigue life region.

Among the specimens whose micro shrinkage volume fraction measured on the fatigue fracture surface was 5–10%, typical five specimens were selected, and then the distributions of the sizes of the micro shrinkage and pore measured on the fatigue fracture surface and on the cross section near the fatigue fracture surface were analyzed. The results are shown in Fig. 10. In this case, also, the sizes of micro shrinkage and pore that have the size of over 10 \(\mu m\) were measured because the micro shrinkage and pore with a size of below 10 \(\mu m\) were difficult to distinguish from dimples on the fracture surface in the case of the measurement on the fractographs. The number and size of the micro shrinkage that has the size over 10 \(\mu m\) measured on the fatigue fracture surface are greater as compared with those measured on the cross section near the fatigue fracture surface. Therefore, the fatigue crack propagates with preferentially linking the micro shrinkages. The number and size of the pore measured on the fatigue fracture surface is nearly equal to those measured on the cross section near the fatigue fracture surface. Therefore, the effect of the pore on the fatigue properties is much smaller as compared with the micro shrinkage.

The typical distributions of the sizes of the micro shrinkage over 10 \(\mu m\) measured on the fatigue fracture surface and on the cross section near the fatigue fracture surface in the low cycle fatigue life region and in the high cycle fatigue life region are shown in Fig. 11. In the case of the specimen broken in the low cycle fatigue life region, the difference in the number of the micro shrinkage measured on the fatigue fracture surface and on the cross section near the fracture surface is large, but in the specimen broken in the high cycle fatigue life region, the difference in the numbers of the micro shrinkage measured on the fatigue fracture surface and on the cross section near the fracture surface is small. Therefore, the trend for the fatigue crack to propagate with linking the micro shrinkage is much stronger in the low cycle fatigue life region where the applied stress is higher than in the high cycle fatigue life region where the applied stress is low. The effect of the micro shrinkage on the fatigue strength is greater in the low cycle fatigue life region than in the high cycle fatigue life region. The fatigue crack initiated from the largest micro shrinkage in both low cycle fatigue life region and high cycle fatigue life region as indicated by arrows in Fig. 11.

Then, the relationships between \(K_I_{\text{max}}\), calculated using
Fatigue Properties of Cast Ag–Pd–Cu–Au–Zn Alloy for Dental Applications in the Relation

the eq. (1) where the micro shrinkage that becomes to be crack initiation site is assumed as the initial crack, and it is approximated as ellipse and the number of cycles to failure obtained in Non-polished and Polished cast specimens are shown in Fig. 12. The scatter of the data is smaller in both cast specimens when the fatigue life is related with $K_{I \text{max}}$ than when the fatigue life is related with the maximum cyclic stress shown in Fig. 6. As stated in the fracture surface observation result, the micro shrinkage controls the fatigue strength of both cast specimens.

In general, dental prosthetic materials sustain a stress between 20 and 230 MPa by mastication, and moreover should sustain the cyclic stress over 10,000,000 times ($10^7$ cycles) that is the number of cycles equivalent to the number of the mastication for around ten years. Therefore, in this study, the target value of the fatigue limit of cast specimen was considered to be 230 MPa that is the greatest mastication stress mentioned above. Since the fatigue strength of this cast specimen is strongly dependent on the size of the micro shrinkage that becomes to be the fatigue crack initiation site, it is better to estimate the size of the micro shrinkage that may act as a fatigue crack initiation site and lead to a fatigue limit of 230 MPa, and better to know its tolerable size in order to promote the reliability of the casting against the fatigue fracture. Therefore, the experimental relationship between $K_{I \text{max}}$ and the number of cycles to failure for Non-polished specimen shown in Fig. 12 was curve fitted. Then, the eq. (2) that describes the relationship between $K_{I \text{max}}$ and the number of cycles to failure was derived as follows:

$$K_{I \text{max}} \approx 2.472 \times 10^3 \times N_f^{(-0.236)}$$

(2)

where $N_f$ is the number of cycles to fracture, and $2.472 \times 10^3$ and $-0.236$ are the material physical property values for Non-polished cast specimen.

Subsequently, the eq. (1) was substituted into the eq. (2), and the eq. (3) for evaluating the approximated ellipse area, $C.D.A.$, of the micro shrinkage that acted as a fatigue crack initiation site from the maximum cyclic stress and the number of cycles to failure in Non-polished cast specimen was derived as follows:

$$C.D.A. \approx \{(2.847 \times 10^3 \times N_f^{(-0.236)})/\sigma_{\text{max}}\}^4$$

(3)

where $2.847 \times 10^3$ and $-0.236$ are the material physical property values for Non-polished cast specimen.

In order to satisfy a target fatigue limit value of 230 MPa for Non-polished cast specimen, $C.D.A.$ is found to be needed below $5.8 \times 10^{-9}$ m$^2$ that is equivalent to the area of the micro shrinkage, of which diameter is 80 µm. However, the average value of $C.D.A.$ in this study was $2.0 \times 10^{-8}$ m$^2$, and many specimens did not satisfy this value of $C.D.A.$ Among all cast specimens (Non-polished and Polished cast specimens),
the percentage of the cast specimens that satisfy the C.D.A. below $5.8 \times 10^{-9} \text{ m}^2$, that is, the diameter below 80 µm, was about 20%. Therefore, improvement in this ratio will promote the reliability for the fatigue strength of this cast alloy. It is necessary to develop the casting method that decreases the area of the micro shrinkage that affects the fatigue strength or to improve the fatigue properties of the alloy itself by microstructural control.

4. Conclusions

The relationships between casting defects and fatigue properties of Ag–Pd–Cu–Au–Zn alloy cast using a dental casting machine were investigated in the comparison with the drawn Ag–Pd–Cu–Au–Zn alloy in this study. The following results were obtained.

1) The fatigue strength of the cast Ag–Pd–Cu–Au–Zn alloy is considerably smaller than that of the drawn Ag–Pd–Cu–Au–Zn alloy in both low cycle fatigue life region and high cycle fatigue life region.

2) The fatigue crack of Non-polished and Polished cast alloys initiates at the micro shrinkage near the specimen surface.

3) The scatter of the fatigue strength of the cast alloy becomes to be small by relating the fatigue life with the maximum stress intensity factor calculated assuming the micro shrinkage that becomes to be a fatigue crack initiation site as an initial crack. This means that the size of the micro shrinkage affects the fatigue strength of this cast alloy strongly.

4) The tolerant size of the micro shrinkage that satisfy the target value of the fatigue limit (230 MPa) of this cast alloy is calculated to be below 80 µm using the equation derived in this study, which describes the relationship between the maximum stress intensity factor and the number of the cycles to failure.

REFERENCES