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ORIGINAL ARTICLE

Microstructure analysis of dental castings used in fixed dental prostheses—a simple method for quality control

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Abstract The aim of this study was to evaluate the microstructural quality of noble alloy castings from commercial dental laboratories using the wiping–etching method as a simple method for quality control. In total, 240 castings from two noble alloys (AuAgCuPt and AuPtZn) were taken from a day's production of five different dental laboratories. The casting quality was evaluated by determining the grain size and by assessing the number and size of shrinkage cavities after acidic etching of the alloy surfaces. The AuAgCuPt alloy castings showed an acceptable quality in the microstructural analysis. The results of AuPtZn castings, however, were not satisfactory because 50.8% of the samples showed a remarkably poorer quality compared to the specifications made by the manufacturer. The proportion of the employed reclaimed alloy had no influence on the casting quality when AuAgCuPt alloy was used, but was influential when casting restorations with AuPtZn alloy. When determining the quantity and size of shrinkage cavities, none of the evaluated castings was of such a poor quality that a replacement of the castings had to be considered. The differences in grain size and quantity of shrinkage cavities were reflecting the individual laboratory process rather than the admixture of new/reclaimed alloy. The presented analysis can be used as a simple method for quality control of dental castings.

Keywords Corrosion · Dental casting · Quality control · Reclaimed alloy · Metallography · Biocompatibility

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Introduction

A combination of public media [1], new materials, and techniques have fueled an esthetic cultural revolution [2]. Although an increasing esthetic demand indicates that all-ceramic restorations might be the material of choice in the future [3], problems resulting in a higher failure rate have been reported [4]. In comparison to all-ceramic fixed dental prostheses (FDPs), metal-based FDPs have shown excellent clinical longevity [5]. Additional reasons like habitual parafunctions, economical circumstances, or even just the unavailability of all-ceramic production facilities are still leading to widely used metal-based restorations.

In restorative dentistry, the quality of dental castings plays an important role. It influences decisively the biocompatibility, increases the oral stability, and lastly ensures the long-term restoration of the patient's disrupted chewing function [5, 6]. Terms like "homogeneity," "fine-grained," "microstructure," "inclusions," and "shrinkage cavity" describe not only structural features in solid materials, but are also influential factors for intraoral longevity and biocompatibility [7, 8]. The most important property of a dental alloy is its chemical stability as its degradation might result in adverse cytotoxic reactions to the host tissue [9] and a release of metal ions into the gingival tissue [7], which can lead to esthetical problems due to discoloration [10]. Additionally, the casting process can result in changed wetting and tarnish characteristics [11].

Besides the biocompatibility, physical properties and the microstructure play an important role for the capability of an alloy to serve as a base for a long-term successful restoration [12]. An alloy with a grain size of over 1,000/mm^2 is called fine-grained [13]; with a grain size of 500/mm^2 or lower, the development of a dendritic structure is likely [14]; and if it is below 100/mm^2, a dendritic structure can always be expected [15]. Alloys with a coarse-grained structure are inferior to fine-grained alloys with regard to their intraoral stability [16]. They are associated with a reduced corrosion resistance [11, 17], increased susceptibility for discoloration [11], a greater risk of metal tearing [18], and a lower fatigue resistance [19].

Several factors accompanying the manufacturing process can influence the casting quality. Among these factors are the composition of the alloy [12], the embedding and pinning methods [20], the preheating and casting temperatures [21, 22], the proportion of reclaimed alloy used [23–26], as well as the melting and casting method itself [27, 28]. The ratio between surface and volume of a casting also plays a crucial role [29].

Today, the standard procedure for conducting structural investigations is metallography [30], whereby sectional areas are treated with an appropriate caustic agent. The objective is to visualize the microstructure by producing reflection and contrast differences [31]. If the surfaces under analysis are wiped repeatedly with a carrier containing a caustic agent until an analyzable pattern becomes visible, the procedure is called wiping–etching technique [32].

To reduce costs, dental laboratories are often adding reclaimed alloy to the new alloy; therefore, inevitably, the question arises whether the structural quality of dental restorations is being compromised as a consequence. Because of these concerns, this study evaluated the quality of dental castings consisting of an admixture of new and reclaimed alloy manufactured under regular conditions in commercial dental laboratories using a wiping–etching technique that could be used as a simple method for quality control.

Materials and methods

For this study, two noble metal alloys were selected: an AuAgCuPt alloy (BioMaingold SG, Heraeus-Kulzer, Hanau, Germany) and an AuPtZn alloy (BioHerador N, Heraeus-Kulzer). The composition of the alloys is listed in Table 1. The test samples consisted of 240 castings from a day's production of five different commercial dental laboratories (Lab A–E) and were manufactured according to the typical method of each laboratory (Table 2). Used crucibles were taken for most of the casting procedures and replaced when necessary. Each crucible was used only for one type of alloy.

Forty castings were taken from Lab B–E and Lab A contributed 80 castings equally made of the two tested alloys. Out of the 40 specimens per laboratory and alloy, 30 crowns and 10 FDP pontics were examined. The crown specimens, which included full crowns, telescopic crowns, and crowns for FDPs, were examined on the outer surfaces. The FDP pontics were analyzed on their buccal surfaces. In the case of ceramic veneering, the analysis was conducted before adding the ceramic to the framework. The average percentage admixtures of reclaimed alloy in each laboratory was different: AuAgCuPt alloy—Lab A 68.0%, Lab B 54.8%, Lab D 51.2%; AuPtZn alloy—Lab A 70.3%, Lab C 66.9%, Lab E 59.0%. The castings of the AuAgCuPt alloy were etched with a mixture of potassium cyanide (10%) and ammonium peroxydisulfate (10%) in a ratio of 1:1 [33]. For the AuPtZn alloy, the specimens were wipe-etched with a mixture containing 30 ml of hydrochloric acid at 37%, 7.5 ml of nitric acid at 65%, 7.5 ml of glacial acetic acid, and 7.5 ml of H_2O [34]. The etching solutions were applied with cotton pellets to a flat area of the specimen to reveal the microstructure in an area with a minimum diameter of 0.5 mm^2. After a 5-s etching period, the etched surface was washed with distilled water and the etching pattern was checked with a light microscope. If the etching was insufficient to evaluate the microstructure, the etching procedure was repeated in 5-s intervals until an adequate etching pattern became visible. The required etching time was recorded.

Table 1 Composition of the noble alloys [in weight percent]

	Ag	Au	Pt	Pd	Cu	Zn	Ir	Mn	Ru	Ta	Grain size
AuAgCuPt alloy	12.3	71.0	4.0	–	12.2	0.5	0.1	–	–	–	500–1,000/mm^2
AuPtZn alloy	–	86.2	11.5	–	–	1.5	–	0.1	0.4	0.3	1,500–2,000/mm^2

Table 2 Casting methods of the dental laboratories

	Casting appliance	Melting method	Casting method
Lab A	Combilabor CL-G 77 (Heraeus, Germany)	Resistance casting	Vacuum–pressure casting
Lab B	Combilabor CL-IG (Heraeus, Germany)	Induction casting	Vacuum–pressure casting
Lab C	Tiegelschleuder TS3 (Degussa, Germany)	Resistance casting	Sling casting
Lab D	Motorcast (Degussa, Germany)	Flame casting, propane/oxygen	Sling casting
Lab E	Drucomat (Wiedeland, Germany)	Resistance casting	Vacuum–pressure casting

The microstructure was evaluated with a stereomicroscope (Leitz CMM, Leitz, Wetzlar, Germany) at a magnification of ×200. The grain size in each casting sample was determined using a modified version of the circle method [32]. For this method, a photograph of the alloy surface and a ruler had to be taken together (Contax 167MT, ×200) and the diameter of the circle was calculated. The circular surface was calculated ($A_C = d^2/4 \times \pi$) and all grains *inside* the circle were counted. Of the grains which were *cut* by the line of the circle, 67% were added to the number of grains counted inside the circle. Grains per square millimeter were calculated from the obtained data.

The grain size was compared with a reference casting sample from the manufacturer. In addition, the calculated mean grain size for each laboratory was compared with those given in the manufacturer's specifications for the alloys (Table 1).

The size and quantity of shrinkage cavities were determined from photographs taken at a magnification of ×200 in accordance with the method described for evaluation of the grain size above. Photographs that could not be evaluated, e.g., due to over-etching or images that were partly out of focus, were excluded from the statistical analysis. The quantity of the shrinkage cavities was sorted in 13 subgroups in 1.8-µm steps beginning from 1.8 to 23.8 µm. Shrinkage cavities larger than 23.8 µm were not found.

The data was statistically analyzed using "SPSS for Windows" (Version 11.5, SPSS Inc., USA) at a level of significance of $P \leq 0.05$. The influence of the laboratory factor (encompassing influences of the melting/casting machine/method and the dental technician), the restoration and the proportion of the reclaimed alloy on the grain size, the quantity of shrinkage cavities, and the required etching time was performed with methods of variance analysis (ANOVA and covariance analysis). Additionally, the correlation between grain size, quantity of shrinkage cavities, and proportion of reclaimed metal was calculated using the Pearson correlation coefficient.

Results

Microstructure analysis

AuAgCuPt alloy

The evaluated mean etching time required to reveal an analyzable microstructure was 36 s (n=120; Table 3). The mean grain size of all 120 samples examined was 908±208/mm^2 (as a typical example, see Fig. 1). The grain size of the reference sample was finer with 1,182 grains per square millimeter. For Lab A, the mean grain size was 1,056±173/mm^2; for Lab B, 788±115/mm^2; and for Lab

Table 3 Mean grain size and standard deviation of the AuAgCuPt and the AuPtZn alloy

Laboratory	Grain size in grains per square millimeter		Etching time in seconds	
	AuAgCuPt alloy	AuPtZn alloy	AuAgCuPt alloy	AuPtZn alloy
Reference sample	1,182	997		
Mean	908±208	1,002±113	36	60
Lab A	1,056±173	–	36	–
Lab B	788±115	–	32	–
Lab D	879±226	–	42	–
Lab A	–	996±99	–	54
Lab C	–	936±85	–	48
Lab E	–	1,068±114	–	81

Additionally, the mean required etching time is shown. Decimals have been rounded (n=40 per laboratory)

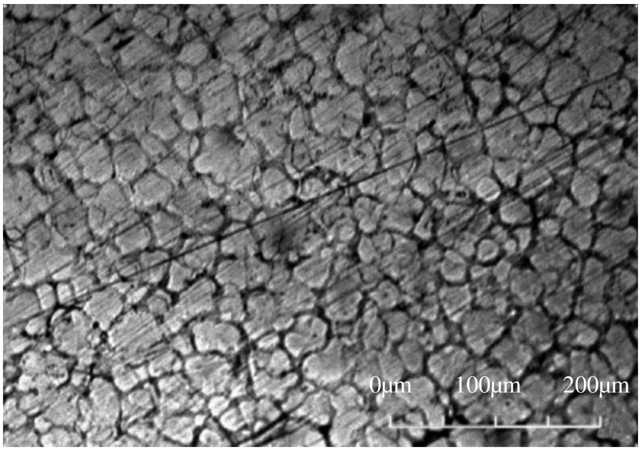

Fig. 1 Microstructure of an AuAgCuPt alloy crown from Lab A with a grain size of 1,334/mm^2

D, 879±226/mm^2 (Table 3). When comparing the grain size of crowns and FDP pontics, crowns always showed a finer-grained microstructure than the pontics in all three laboratories (Fig. 2). The percentage of reclaimed alloy varied between the laboratories (Fig. 3). On average, Lab A used 68%, Lab B used 55%, and Lab D used 51% of reclaimed alloy. However, there was no significant correlation (r=0.058; P=0.5336) between the number of grains and the proportion of reclaimed alloy calculated for all laboratories. The results of the variance and covariance analysis are summarized in Table 4.

AuPtZn alloy

For the AuPtZn alloy, the evaluated mean etching time required to reveal an analyzable microstructure was 60 s (n=120; Table 3). The mean grain size of all 120

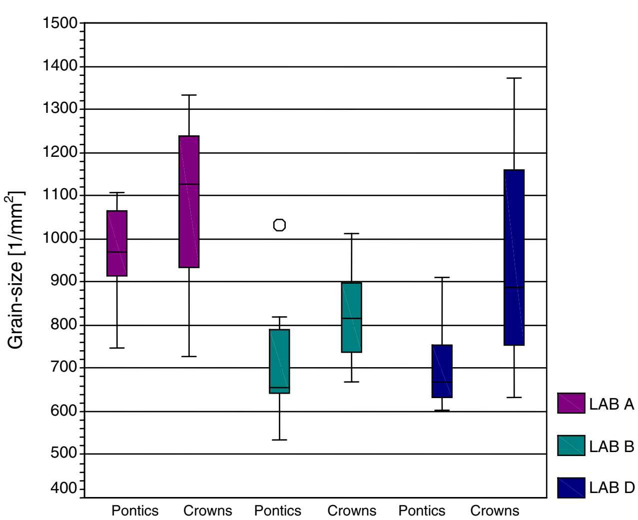

Fig. 2 Comparison of the grain size [1/mm^2] of crowns and pontics from different dental laboratories for the AuAgCuPt alloy

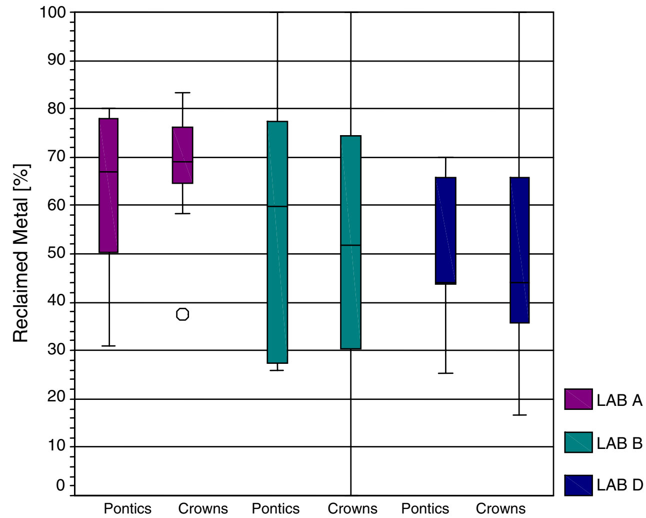

Fig. 3 Comparison of the reclaimed metal of crowns and pontics from different dental laboratories for the AuAgCuPt alloy

samples examined was 1,002±113/mm^2. The grain size of the reference sample was similar with 997 grains per square millimeter. For Lab A, the mean grain size was 996±99/mm^2; for Lab C, 936±85/mm^2; and for Lab E, 1,068±114/mm^2 (Table 3). Comparing the grain size of crowns and FDP pontics, crowns and the AuPtZn alloy always showed a finer-grained microstructure than the pontics in all three laboratories (Fig. 4). Again, the proportion of reclaimed alloy varied among laboratories (Fig. 5). On average, Lab A used 70%, Lab C used 67%, and Lab D used 59% of reclaimed alloy. A significant correlation (r=−0.39; P=0.0001) between the number of grains and the proportion of reclaimed alloy could be revealed for all laboratories. The higher the proportion of reclaimed alloy used, the lower was the exhibited grain size. The results of the variance and covariance analysis are summarized in Table 5.

Quantity and size of the shrinkage cavities

AuAgCuPt alloy

From the overall 120 specimens, 86 were suitable for evaluation (Lab A n=27, Lab B n=25, Lab D n=34). The findings of the shrinkage cavity analysis are illustrated in Fig. 6a, b. Shrinkage cavities for crowns could be found from 1.8 to 20.2 µm and for FDP pontics from 1.8 to 23.8 µm. No correlation between any of the 13 shrinkage cavity sizes and the grain size was revealed (r=−0.35 to 0.22, P>0.05).

Out of the 13 shrinkage cavity subgroups, one-factorial variance analysis revealed a significant dependence on the laboratory for the 1.8-µm cavity size only (P≤0.001). An additional multivariance analysis showed that the quantity of cavities was significantly influenced by the laboratory and the restoration type (P≤0.001).

Table 4 Statistically significant influences on the microstructure of the AuAgCuPt alloy (ANOVA and covariance analysis)

	Laboratory	Restoration	Etching time	Reclaimed metal	Interaction
Grain size	***	***	n.a.	N.S.	Reclaimed metal/laboratory*
Etching time	***	*	n.a.	N.S.	Laboratory/restoration *
Grain size (crowns)	**	n.a.	N.S.	**	Laboratory/reclaimed metal**
Grain size (pontics)	***	n.a.	N.S.	N.S.	N.S.

N.S. not significant ($P>0.05$), *n.a.* not applicable

*$P\leq0.05$, **$P\leq0.01$, ***$P\leq0.001$

AuPtZn alloy

From the overall 120 specimens made of the AuPtZn alloy, 67 were suitable for shrinkage cavity analysis (Lab A $n=21$, Lab C $n=21$, Lab E $n=25$). The findings of the shrinkage cavity analysis are illustrated in Fig. 7a, b. Shrinkage cavities for crowns ranged from 1.8 to 22.0 µm and for FDP pontics it ranged from 1.8 to 23.8 µm. Only the 16.5-µm cavity size out of the 13 shrinkage cavity size subgroups showed a significant correlation to the grain size ($r=-0.35$, $P=0.004$).

Shrinkage cavities sized 5.5, 7.3, 9.2, and 16.5 µm were significantly influenced by the laboratory ($P\leq0.05$). The additional multivariance analysis showed a significant influence of the laboratories and the restoration type ($P\leq0.05$).

Comparison of the shrinkage cavities in the AuAgCuPt alloy and the AuPtZn alloy

For AuAgCuPt alloy, the lower frequency of shrinkage cavities in comparison to the AuPtZn alloy of the size categories 1.8, 3.6, 5.5, 7.3, 9.2, 11.0, 12.8, and 16.5 µm was significant ($P\leq0.05$).

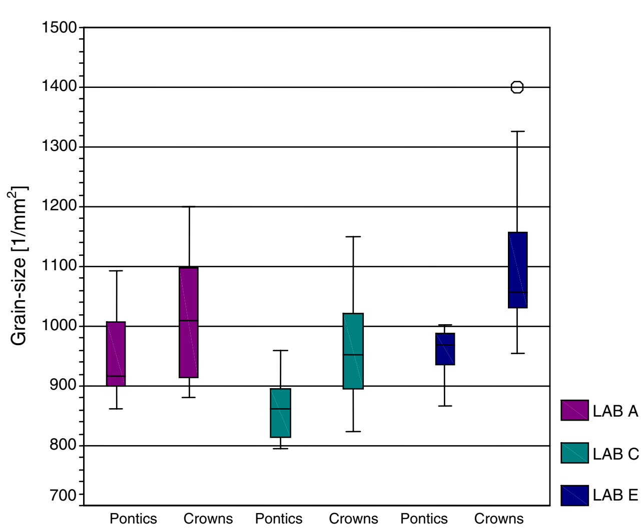

Fig. 4 Comparison of the grain size [1/mm^2] of crowns and pontics from different dental laboratories for the alloy AuPtZn alloy

Discussion

The aim of this study was to evaluate the casting quality in commercial dental laboratories with a wiping–etching technique and whether this method is suitable as a simple method for quality control in the dental laboratory. The specimens were subjected randomly to a microstructural analysis, which determined the grain size and the quantity and size of shrinkage cavities. This approach tested the practical applicability of the method and the utility of this simple method for dental technicians and dentists [34, 35]. Etchants are commonly used in dentistry to alter alloy surfaces for evaluation purposes [36] or restorative reasons [37]. The only prerequisite for the wiping–etching technique is the production of the appropriate caustic agent for the respective alloy at a correct concentration. Since no significant correlation between etching time and grain size could be found, the application of the wiping–etching method may represent a potential technique allowing dental technicians and dentists alike to selectively examine the quality of casted restorations.

The procedure is well-suited for determining the grain size and—with some limitations—for the determination of the quantity and size of shrinkage cavities. However, clearly defined criteria for acceptable and unacceptable castings have not yet been established. According to a recommendation by the German Federal Health Agency (Bundesgesundheitsamt) [38], a dental casting should not be used if it contains a shrinkage cavity with a diameter of over 100 µm, i.e., a shrinkage cavity that is visible by the naked eye. A quality control with the wiping–etching technique could help to state more precisely guidelines for acceptability of dental casted restorations and could be used for in vitro testing before clinical usage of dental alloys.

In the case of the AuAgCuPt alloy, the average microstructure of the castings did not meet the criterion for a fine-grained microstructure, i.e., a grain size of over 1,000/mm^2 [13]. The grain size of the reference sample casted by the manufacturer under laboratory conditions (1,182 grains per square millimeter) was finer than the average grain size of the castings from the commercial laboratories (908 grains per square millimeter).

However, differences between the laboratories could be found. Lab A showed, on average, a fine-grained micro-

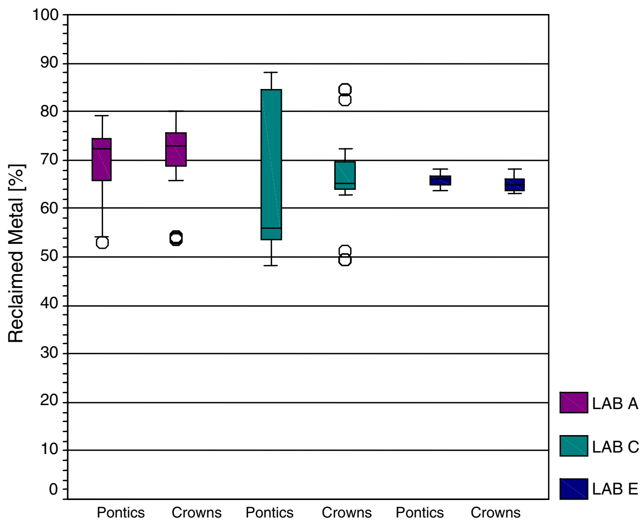

Fig. 5 Comparison of the reclaimed metal of crowns and pontics from different dental laboratories for the AuPtZn alloy

structure, whereas the average microstructure produced in Lab B and D was classified as coarse-grained [13]. For Lab A, 60% could be classified as fine-grained, whereas in Lab B 95% and in Lab D 70% of all samples ranged between 500 and 1,000 grains per square millimeter and thus exhibited a coarse-grained microstructure. For the AuPtZn alloy, the average microstructure of all cast samples barely conformed to the definition of fine-grained [13]. For Lab A, 55% and, for Lab C, 75% of all samples exhibited a coarse-grained structure. In contrast, for Lab E, almost 73% of the castings could be classified as fine-grained. Overall, the grain size of the castings made from the AuAgCuPt alloy ranged within or above the specifications provided by the manufacturer (Table 1), while none of the castings made of the AuPtZn alloy was in this range.

In the search of possible explanations, the admixture of reclaimed alloy could have been influential. The use of sometimes up to 100% reclaimed alloy for casting dental restorations differs considerably from the recommendations in the literature [23], which are in the range of one third to a maximum of two thirds reclaimed metal for noble alloys. To recast a noble alloy several times may result in changed physical characteristics [25]. However, in contrast to other studies [39, 40], in this study, a high admixture of reclaimed alloy in AuAgCuPt castings did not result in a significant reduction of the grain size for all laboratories, whereas the AuPtZn alloy showed a significant correlation between a higher admixture of reclaimed alloy and a degraded grain size. With the limitation of different alloys and casting methods, studies report contrasting results with regards to physical properties, such as corrosion resistance, surface properties, or tensile strength [26, 40, 41]. One study even states an increase in corrosion resistance after remelting and recasting [25].

In the current study, a significant effect of the laboratory factor on the casting quality was found. It has to be taken into account that the laboratory factor includes a combination of factors like casting machine-, casting method-, and operator-dependent influences. Due to the different melting methods [28], casting methods [41], and dental technicians in each laboratory, the influence of single factors on the casting quality could not be evaluated separately.

Besides the laboratory factor for the AuPtZn alloy, the admixture of the reclaimed alloy seems to have played a decisive role in each laboratory. Both facts might lead to the suggestion of a quality control for the casting process to reduce physical [12] or esthetical [10] failures.

Crowns always exhibited a finer-grained microstructure than FDP pontics. For the AuAgCuPt alloy, 63% of the crowns but only 17% of the pontics were fine-grained. For the AuPtZn alloy, 59% of the crowns and also 17% of pontics were fine-grained, suggesting that the ratio between surface and volume of a casting influences its microstructure. With the smaller surface/volume ratio of the FDP pontics, the cooling process takes more time [42]. During the same time period, as the alloy solidifies, fewer nuclei for crystallization form in the pontics. The microstructure, therefore, becomes more coarse-grained. In the crowns, the atoms lose their kinetic energy faster and, therefore, more nuclei for crystallization form. For this reason, FDP pontics require a more critical evaluation in terms of their grain size than thin-walled crowns.

Similar to the microstructural analysis, a comparative assessment of the quantity and size of shrinkage cavities with other studies is only possible to a limited extent. For

Table 5 Statistically significant influences on the microstructure of the AuPtZn alloy (ANOVA and covariance analysis)

	Laboratory	Restoration	Etching time	Reclaimed metal	Interaction
Grain size	***	***	n.a.	***	N.S.
Etching time	***	N.S.	n.a.	N.S.	N.S.
Grain size (crowns)	**	n.a.	N.S.	***	N.S.
Grain size (pontics)	***	n.a.	N.S.	***	N.S.

N.S. not significant ($P>0.05$), *n.a.* not applicable

*$P\leq0.05$, **$P\leq0.01$, ***$P\leq0.001$

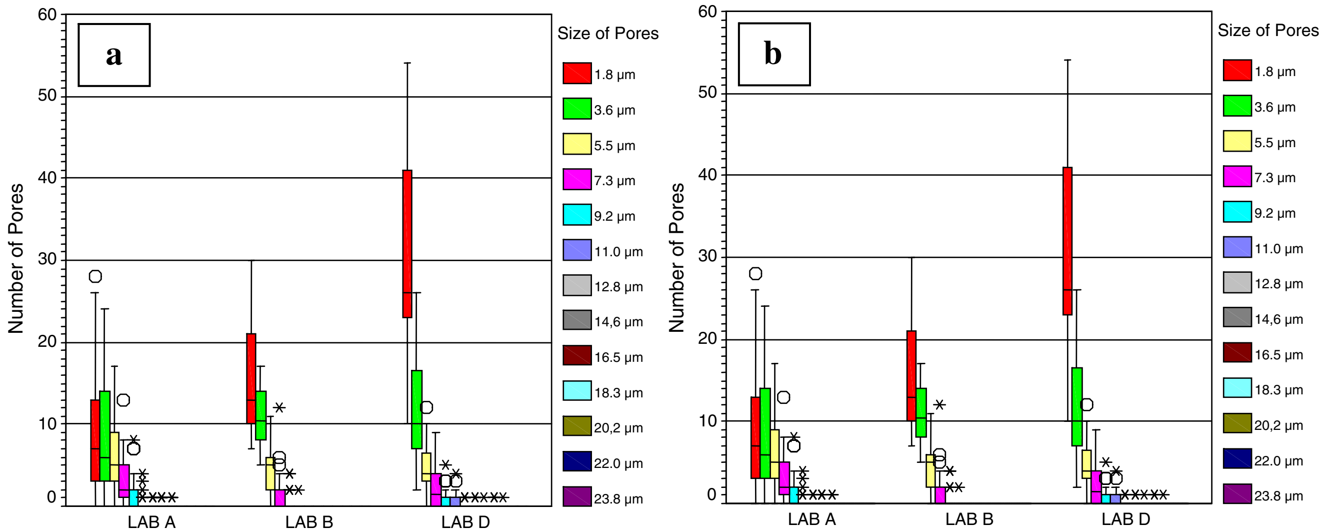

Fig. 6 Comparison of the quantity of the shrinkage cavities made of AuAgCuPt alloy **a** for crowns and **b** for FDP pontics from different dental laboratories

instance, one study [43] determined only the largest shrinkage cavity per surface or casting object, respectively. Shrinkage cavities occur mainly in the dendritic interspaces of the casting microstructure. As a result, the finer the grain size and consequently the smaller the interspaces are, the less likely the tendency to develop shrinkage cavities [23]. However, in this study, no general relationship between the grain size and the occurrence of shrinkage cavities was observed. The increased occurrence of shrinkage cavities in FDP pontics for both alloys might be related to the differences in the volume of crowns and pontics. The greater the volume of a casting, the more difficult it is for the dental technician to choose the pinning method to predetermine correct solidification direction. However, only a correct assessment of the solidification direction can minimize the occurrence of shrinkage cavities [23].

The significantly higher values ($P \leq 0.0001$) for the quantity and size of the shrinkage cavities from Lab D, which used a flame-melting method confirms again the fact that flame-melting comprises a higher risk for a reduced casting quality [23, 44]. However, due to a limitation in the study design, we could not evaluate the specific influence of the casting methods on the formation of shrinkage cavities. Nevertheless, different casting methods seem to not inevitably result in a different casting quality [28]. Similar to the grain size findings, possible reasons for the different quantity of shrinkage cavities might be found in the laboratory factor and the surface/volume ratio.

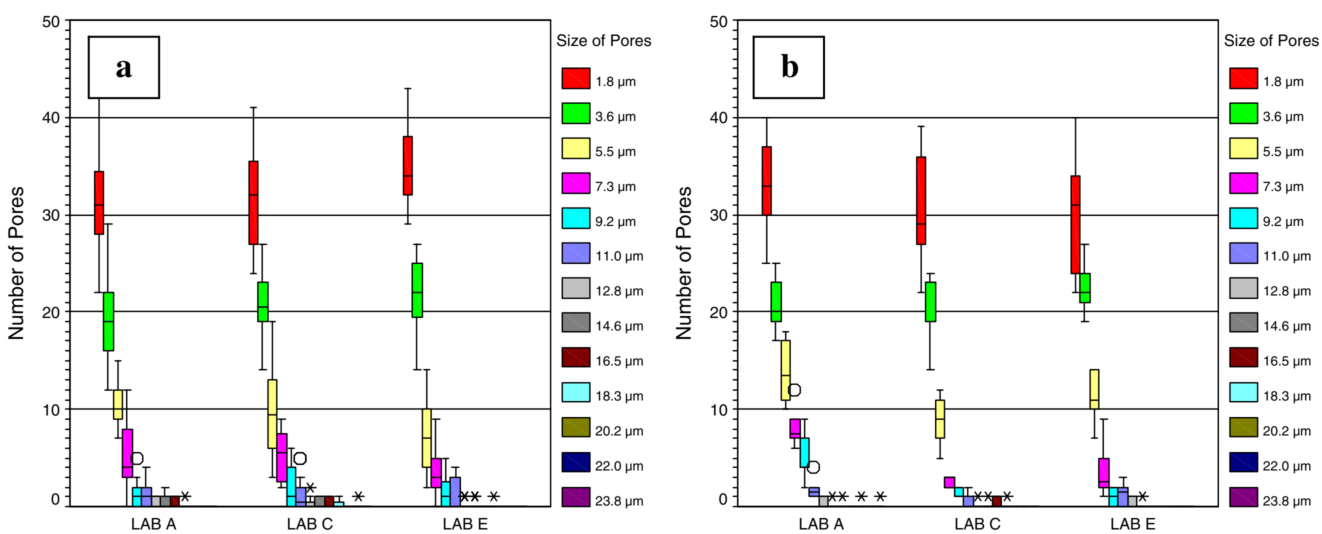

Fig. 7 Comparison of the quantity of the shrinkage cavities made of AuPtZn alloy **a** for crowns and **b** for FDP pontics from different dental laboratories

Of the two alloys evaluated in this study, not a single shrinkage cavity was found that exceeded 23.8 µm. Therefore, according to the guidelines of the German Federal Health Agency [38], none of the casting samples required a remake. Most of the shrinkage cavities occurred in the size categories 1.8, 3.6, and 5.5 µm and were localized. The surface polish of these castings was acceptable, and thus, these porosities are not clinically relevant [14].

The significant differences in the quantity of shrinkage cavities between the two alloys appear to be reflected in their respective physical properties. Particularly interesting in this context is the percentage of elastic elongation. The AuAgCuPt alloy has a value of 16%, which is significantly higher than that of the AuPtZn alloy at 6%. A substantial elastic elongation indicates a highly ductile material, while a smaller elastic elongation indicates a brittle material [23]. The smaller elastic elongation of the AuPtZn alloy is apparently the consequence of the larger quantity of small shrinkage cavities. Trimming and polishing by the dental technician can eliminate small shrinkage cavities as long as the alloy is able to undergo sufficient elongation that the polish causes. But polishing cannot remove larger shrinkage cavities; the material will exceed its elastic elongation limit before the large cavity cross-section is polished completely [14].

Conclusions

Under the limitations of the study, the following conclusions can be drawn:

- The wiping–etching method is a simple method to control the quality of dental castings in dental laboratories.
- The flame-melting method should not be used for casting dental restorations, since it had a significant negative effect on the quantity and size of shrinkage cavities.
- The quantity and size of the shrinkage cavities was significantly dependent on the laboratory factor and the volume/surface ratio of the casting.
- When using the AuAgCuPt alloy for dental castings, the proportion of reclaimed alloy was not as influential on the grain size as the laboratory factor and the volume/surface ratio of the casting.
- When using the AuPtZn alloy for dental castings, a higher proportion of reclaimed alloy influenced the grain size negatively. Additionally, the volume/surface ratio and the laboratory factor showed an impact on the grain size.

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Conflict of interest The authors declare that they have no conflict of interest.

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