Powder data file as a tool for identification of dental gold alloys

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In order to obtain data for a prospective powder data file for dental gold alloys X-ray diffraction photograms of 75 different gold alloys were taken using a Guinier-Hägg camera and CuKα_1 radiation. Before the X-ray photograms were taken the alloys had been heat treated at 700 °C, 800 °C or 900 °C depending on the solidus temperatures of the alloys. The lattice parameters of the samples were determined from the X-ray photograms. These data were supplemented by a specification of the interplanar spacings of the three strongest lines on the diffraction patterns and also by the relative intensities and Miller indices of these lines. An extensive library of reference films is to be collected and this and other details of the file are discussed. It is not intended that the file be used to identify a particular alloy but merely to identify the type of alloy. Thus it can serve as a guide to facilitate the proper selection of gold alloys within practical clinical dentistry.

Key-words: Dental gold alloys; lattice parameter; dental materials

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The rapidly increasing number of varieties of dental gold alloys for crown and bridge prostheses has led to a situation where it very often is desirable to identify the type of alloy or alloys used in a certain fixed construction cemented earlier. It is necessary to avoid an unknown mixture of gold alloys within the oral cavity because of the physical properties of the constructions as well as the risks of oral galvanism. However, it is almost impossible for the dentist to make an *in situ* identification of the alloys used for example in a bridge which was cemented many years ago by another dentist. Any method which would facilitate the rapid identification of the alloy or alloys used in a fixed dental gold construction would therefore be of a great value.

X-ray powder diffraction analysis allows the identification of the type of alloy or alloys in question using a very small amount of alloy material. With a dental coromant bur some grains of the alloy can easily be scraped from the *in situ* construction. The amount of material needed, about 100 μg, is similar to that removed during a very slight grinding.

The diffraction pattern given by a crystalline substance is unique because the positions of the reflections are determined by the spacing between the layers of atoms in a crystal and the intensity of these reflections is

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dependent on the arrangement of the atoms in the crystal lattice. The simplest way to identify a single substance is to compare its diffraction photogram visually with those of known materials taken under identical conditions. This implies that an extensive library of reference films from dental gold alloys must be available for such comparisons (4). The X-ray powder photograms cannot be used to prove the existence of small differences in compositional additives but this is unnecessary in terms of intended use within practical clinical dentistry. According to a standard work such as the powder data file of inorganic compounds published 1973 by National Bureau of Standards (6) the lattice parameter of pure gold is 4.07897 ± 0.00007 Å at 25 °C.

Thus the aim of the present work was to begin a powder data file initially for those dental gold alloys which Scandinavian dentists are expected to use. However, it is the intention that this data file is continuously extended and completed until it becomes a tool for routine identification of the type of alloy.

MATERIALS AND METHODS

75 different commercial dental gold alloys both for casting and for soldering operations were included in the present study (Table I). The alloys which were of the types intended for crown- and bridge-work using the conventional technique as well as the porcelain-fused-to-metal technique were bought on the open market.

Each alloy sample was melted and quenched. Thereafter each specimen was heat treated for 6 hours at 700 °C (solder alloys), 800 °C or 900 °C (casting alloys) and again quenched. The difference in temperature during heat treatment was due to *the range of the solidus* temperatures of the alloys in question. Powder specimens were prepared using a dental coromant bur. To reduce the effects of lattice distortions occuring because of plastic deformation during powder preparation each powder specimen was heat treated for 5–10 minutes, also at 700 °C, 800 °C or 900 °C respectively, to allow recrystallisation but no sintering of the powder. Thereafter the powder specimens were quenched. All the meltings and heat treatments were carried out in an electrically heated furnace with a controlled atmosphere (argon, AGA SR). The specimens were put separately in to crucibles of recrystallized alumina and placed in the furnace which was already at the required temperature.

Using a Guinier-Hägg camera and CuKα_1 radiation ($\lambda = 1.54051$ Å) X-ray powder diffraction photograms of all the specimens were taken. Silicon, which has a unit cell edge of 5.43088 Å at 25 °C (2), was used for calibration of the camera constant. The lattice parameters $a \pm \sigma a$ were refined by a least-squares treatment of the $\sin^2\theta$ values using a computer program, PIRUM, developed by Werner (5).

RESULTS

Line broadening due to lattice strain and segregations was avoided or reduced as a result of the annealings carried out before the X-ray photograms were taken. Thus the positions of the sharp lines could be determined with an accuracy of 0.01 mm. The corresponding uncertainty of the lattice dimensions is about 0.1 ‰ (3). The structures observed on all the photograms were face-centered cubic. However, certain very weak extra lines were observed on some of the photograms. As these weak lines were not attributable to traces of impurity they indicate a tendency towards ordering.

In addition the photograms of some of the newest alloys contained sharp lines from two different phases. Thus the alloys Degulor NF IV, Stabilor NF IV and Novostil each contained two face-centered cubic structures with different cell edges. Experimental data are collected in Table 1, where the lattice parameter, a, with standard deviation σa, is given.

DISCUSSION

Different atom positions in the metal lattice are specified in a common co-ordinate system where the three axes are parallel to the edges of the unit cell. Lattice planes are defined in terms of the so called Miller indices (hkl) which are the reciprocals of the intercepts of the planes on the coördinate axes. For the cubic system the following relationship between cell dimension a and the interplanar spacing d is:

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad (1)$$

If this equation is combined with the Bragg equation:

$$n\lambda = 2\, d \sin \theta \quad (2)$$

where n, the order of diffraction, is an integer which by convention is incorporated in the indices of the plane, λ is the wavelength of the X-rays and θ is the angle of reflection, we obtain:

$$\sin^2 \theta_{hkl} = \frac{\lambda^2}{4a^2} (h^2 + k^2 + l^2) \quad (3)$$

Depending on the type of lattice certain reflections will be systematically absent. The reflection condition for the face-centered cubic system implies that hkl reflections are present only when h, k, l are all odd or all even (3). The alloys studied in the present work gave reflections for the (hkl) planes (111) (200) (202) (311) (222) (Fig. 1).

In the present work difficulties occasionally occurred in obtaining the optimum crystallite size which is about 10^{-4} cm. Larger crystallites give broken or spotty powder lines because the volume of the sample irradiated does not contain sufficient randomly orientated crystallites. Those smaller than 10^{-4} cm cause pure diffraction broadening beyond that expected from for example lattice distortions or compositional variations. In these cases new powder specimens were prepared from the same original sample using different sized dental coromant burs until powder patterns of sufficient sharpness were attained. The powder particles could also be passed through a sieve. As stated earlier line broadening due to compositional variations, i.e. segregations, and to lattice strains following distortion were kept under control by annealings.

The powder patterns from most of the dental casting gold alloys which are in use at present contain only one phase and the patterns can be indexed in terms of a face-centered unit cell. It is remarkable therefore to find that some of the newest casting alloys contain two phases, both of which have a face-centered cubic symmetry. For example the alloys Degulor NF IV and Stabilor NF IV are both free from copper, a fact which has been given great publicity by the manufacturer. Copper contributes to the strength and hardness of gold alloys and takes part in their age hardening (1). As might be expected the heterogeneous structure of these new two-phase dental gold alloys offers an increased resistance to slip. Thus an attempt has been made to compensate for the removal of copper. Nevertheless, to make the alloys less homogeneous in this way can be unsuitable with respect to conceivable effects on corrosion resistance. Findings of this kind strongly emphasize the importance of a continuous control of dental gold alloys. As can be seen from Table 1 there are certain alloys, for example Degulor M, Duallorlot 2, Dentallod 1 and ANA CD which have the same or almost the same value for their lattice parameter. As the X-ray powder photograms cannot be used

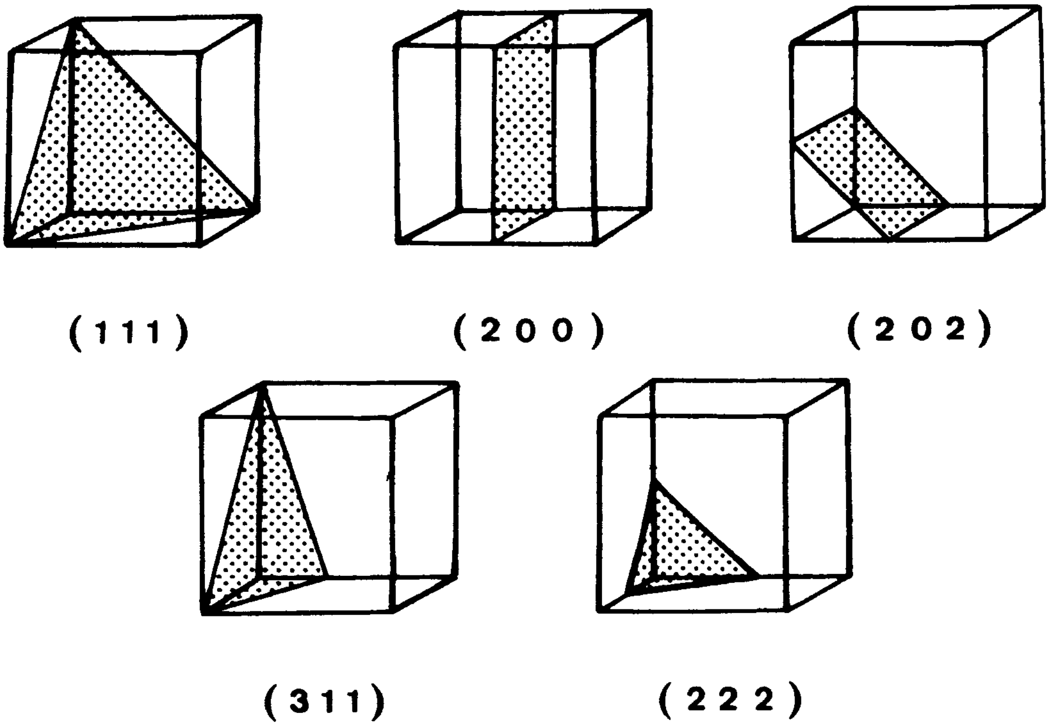

Fig. 1. The lattice planes, with corresponding Miller indices, of the face-centered cubic cell from which reflections were obtained.

to prove the presence of small compositional additives many solutes of gold alloys of adjacent compositions will produce the same value for the lattice parameter. Thus the unit cell edge alone cannot be used for final identification of the alloy. To improve the possibilities of differentiation the diffraction pattern of the alloy to be identified can be compared directly with those from known alloys with the same cell edge. Great differences in the type of alloy and thus in the lattice parameter imply an increased risk of oral corrosion due to differences between the alloys as regards the tendency towards oxidization.

Data concerning crystal symmetry and lattice parameter as presented in Table 1 of the present work have been supplemented by a specification of the interplanar spacings of the three strongest lines on the diffraction patterns as calculated from eqn. (1) and also by the relative intensities and Miller indices of these lines. The relative intensities of the lines were estimated by visual inspection in a projector. In Table 2 an example is given of how these supplementary data have been listed. Remarks on how the information was obtained, crystallographic, optical and physical data, the source of the material and the manner of its preparation have also been noted. Finally all data and information regarding an alloy are collected on a card; one card for each alloy. In this way a card index will be obtained and it should be possible to develop the powder data file as a tool for routine identification of the type of alloy. Thus the file can be used as a guide to facilitate the proper selection of gold alloys within practical clinical dentistry. It is the intention that the data register as well as the library of reference films will be continuously extended in order to improve the possibilities of rapidly identifying the type of any dental gold alloy.

Table 1. *Cell dimension* $a \pm \sigma a$ *of the face-centered cubic unit cell of the alloys studied*

Manufacturer	Commercial name of the alloy	Unit cell dimension, a in Å	$\sigma(a) \cdot 10^3$ in Å
AB Nordiska affineriet Hälsingborg, Sweden	ANA A	4.038	0.3
	ANA B	4.065	0.3
	ANA C	3.978	0.3
	ANA CD	3.981	0.3
	ANA D	4.039	0.4
	ANA E	3.921	0.1
	ANA lod 820	3.942	0.2
	ANA lod 850	3.952	0.1
AB John Sjöding Solna, Sweden	JS B	4.061	0.1
	JS C	3.970	0.3
	JS C3	3.972	0.1
	JS D	3.952	0.1
	JS D2	4.011	0.4
	JS E	4.016	0.3
	JS E2	3.966	0.5
	JS M	4.050	0.3
	JS V	3.945	0.2
	JS 18 karat	3.970	0.2
	JS 20 »	4.069	0.3
	JS 22 »	4.073	0.2
	JS 23 »	4.075	0.2
	JS 24 »	4.076	0.1
	JS Hårdlod 730	4.001	0.2
	JS » 820	3.964	0.1
	JS » 850	3.969	0.0
Heraeus Hanau, Germany	Herador	4.048	0.3
	Herador Lot II	4.005	0.5
Howmedica Int. Köln, Germany	Microbond lod	4.043	0.1
Degussa Frankfurt, Germany	Degulor C	4.026	0.4
	Degulor M	3.981	0.1
	Degulor MO	4.042	0.2
	Degulor NF IV	3.951, 4.058	0.2, 0.3
	Stabilor NF IV	4.069, 4.015	0.3, 0.2
	Primallor G	3.949	0.3
	Degudent	4.043	0.2
	Degudent G	4.064	0.1
	Degudent N	4.043	0.3
	Degudent U	4.029	0.1
	Degulor-Lot 1	4.006	0.2
	Degulor-Lot 2	3.983	0.3
	Duallor-Lot 1	3.972	0.7
	Duallor-Lot 2	3.980	0.3
	Degudent-Lot U 1	4.052	0.4
Cendres & Metaux, Biel/ Bienne, Suisse	Protor-Lot 1	3.965	0.3
	Protor-Lot 2	3.962	0.1

Metaux Precieux Neuchatel, Suisse	Pontor 2	3.967	0.3
	Aurofluid 2	4.054	0.3
	Aurofluid 3	3.943	0.4
	V 4	4.046	0.4
	V 44 Dur	4.024	0.1
	V-Super	4.048	0.3
	V-Delta	3.992	0.4
	Novostil	3.998, 3.915	0.2, 0.1
	V-Delta hartlot	4.041	0.2
	Pontor hartlot	3.928	0.1
	Pontor weich lot	3.992	0.3
	V-weich lot	3.963	0.5
	V-weich lot (without Cd)	3.975	0.2
	V-hart lot	4.067	0.1
	Vacu-V-lot	3.986	0.3
	Standard 1	3.956	0.4
	Standard 2	3.974	0.4
	Standard 3 (without Cd)	3.975	0.2
Ädelmetall AB Malmö, Sweden	Auro-platin I	3.993	0.4
	Auro-platin II	3.953	0.3
	Auro-platin III	3.985	0.2
	Aurodent C	3.989	0.3
	Aurodent D	3.946	0.2
	Kronguld	3.984	0.3
	Allguld I	3.987	0.1
	Allguld II	3.972	0.4
	Dentallod nr 1	3.980	0.3
	Dentallod nr 2	3.963	0.2
Jelenko New Rochelle, N.Y. USA	Midas	4.008	0.3
	Jelenko-0	4.047	0.3

Table 2. *Interplanar spacings d, relative intensities I/I_1 and Miller indices hkl, corresponding to the three strongest reflections on the diffraction photogram of the alloy V-Super*

d Å	I/I_1	hkl
2.34	100	111
2.02	70	200
1.43	65	202

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