Metallographic Techniques For Beryllium Copper and Beryllium Nickel Alloys

by

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and

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Beryllium copper and beryllium nickel alloys can be easily examined metallographically by following standard procedures. These procedures are outlined in detail in this paper and provide the information necessary for examining the structure of these alloys. This paper describes procedures for selecting a metallographic specimen; mounting, grinding and polishing the specimen; and etching it to reveal the metallurgical structure. The included photomicrographs serve as examples of the structure of commonly used tempers of these commercial alloys.

Health and Safety

Despite low concentrations of beryllium in commercial beryllium copper and beryllium nickel alloys (nominally 2 wt% or less), these materials can be hazardous to health if excessive quantities of dusts, mists, or fumes containing particles of alloy small enough (10μm or less) to enter the lungs are inhaled. Special precautions are not required for metallographic sectioning, grinding, or polishing performed wet with water or cutting oil or for dry shearing of clean, thin-section strip or wire. Adequate ventilation should be provided for dry sectioning, grinding or polishing operations which produce dust or fumes. Metallographic preparation equipment and laboratory work surfaces should be damp wiped periodically to remove accumulation of dry alloy particles. Beryllium-containing alloys are not harmful in contact with skin or wounds, or if swallowed. For additional information, see the article "Toxicity of Metal Powders" in Volume 7 of the 9th Edition of the ASM *Metals Handbook*. Conventional, commercially available metallographic preparation facilities normally can be used safely to prepare these alloys.

Specimen Preparation

Metallographic equipment and procedures for beryllium-containing alloys are much the same as those recommended for general metallurgical laboratory use.

Sectioning of specimens is carried out by sawing, abrasive cutting, or shearing, depending on section thickness and strength. Abrasive wheels formulated for nonferrous or medium-hardness materials and general purpose use are satisfactory for beryllium copper. Wheels formulated for hard materials and heavy sections are better for beryllium nickel. Abrasive cutting should be performed wet to avoid thermal damage to the specimen and to minimize metallic dust. Sufficient surface metal is then removed from the sectioned faces of the specimen by wet rough grinding to eliminate any deformed material introduced during sectioning.

Mounting is usually required for specimens too small to be hand-held while polishing or for those requiring edge preservation. Flat strip or transverse sections of small diameter rod and wire may be stacked and gripped in reusable metal screw clamps for unembedded preparation. Alternatively, samples may be embedded with cold-mounting or compression-molding resins. Commercial metal or plastic sample clips are used to stand the sample upright in the mold. Transparent mounting resins are preferred for delicate fabricated parts, such as electrical contacts, to help locate features of interest in the final plane of polish. When distortion of the sample under pressure is to be avoided, cold-mounting is favored over compression molding.

Edge retention may be enhanced by nickel plating prior to mounting or by using hard compression-molding resins formulated for edge preservation. Glass beads or alumina (Al_2O_3) granules added to cold-mounting resins for the same purpose will likely contaminate the polishing wheel and cause undesirable specimen scratching, particularly in the softer forms of beryllium copper.

Grinding. Coarse grinding is performed wet on a belt or disk grinder using 120 or 180 grit abrasive paper to remove any deformed metal layer. Fine grinding is also performed wet, either by hand on strips of abrasive paper or mechanically on 300 rpm or faster disks using, successively, 240, 320, 400 and 600 grit abrasives. The sample is rotated 90° between each grinding. Silicon carbide or Al_2O_3 abrasives may be used.

Mechanical polishing is usually accomplished in rough and final stages. Rough polishing is performed using a $6\mu m$ diamond on a wheel covered with a hard, napless chemotextile cloth. Extender oil is applied sparingly, and the wheel is rotated at approximately 300 rpm or less. The specimen is initially positioned so that the direction of polishing is perpendicular to the 600 grit grinding scratches. Maintaining this orientation, it is briefly rotated counter to the rotation of the wheel to distribute the rough polishing scratches randomly.

Final polishing is performed using $0.05\mu m$ Al_2O_3 in distilled water suspension and a wheel covered with low-nap rayon cloth. Speeds are the same as those used for coarse polishing. The wheel is kept moderately saturated with polishing suspension, and the specimen is counter-rotated to vary the direction of final polishing. An exception to this procedure is the case of the softer annealed or lightly cold worked tempers of beryllium copper, in which unidirectional final polishing parallel to the specimen long axis helps to minimize scratching. The specimen is washed under running water after each polishing step with mild soap and a cotton swab, then rinsed with alcohol and dried under a warm air blast.

Automatic Grinding and Polishing. Several automatic metallographic preparation machines are available that provide rapid and reproducible grinding and polishing of multiple specimens through the use of preset pressure control and a cycle timer. These machines use metal sample holder disks that accommodate 4 to 12 or more cylindrical metallographic mounts or various numbers and sizes of unembedded samples. The holders are rotated by the sample mover head of the machine at approximately 150 rpm and are mechanically pressed against the rotating work wheel, which can be a coarse grinding stone, a wheel accepting successively finer grades of abrasive paper, or a cloth covered polishing wheel. Some systems also employ lapping techniques. Work wheels must be manually changed between preparation steps, but the specimens are never removed from the holder until they are ready to be etched. This preserves a common plane of polish and maintains flatness of the prepared surfaces. Due to the high pressures and short cycle times typically used, these automatic machines increase metallographic laboratory productivity and improve edge preservation and phase retention.

Automatic metallographic preparation techniques vary according to the machine used and materials being prepared, but the following procedures have been successfully used for beryllium-containing alloys and can be adapted to any automatic system.

Holders containing mounted specimens or relatively square cut, unembedded specimens are rough ground on 120 or 240 grit paper and fine ground on, successively, 240, 320, 400 and 600 grit papers. Very uneven, unembedded specimens may require coarse 60 or 80 grit stone or paper grinding to bring all the samples in a holder to a single plane of polish. A copious flow of recirculated water-base coolant is applied to the work wheel during each grinding step. It is not necessary to wash the samples in the holder between grindings. Wheel speeds of 150 rpm, pressures of 150 N (35 lbf), and times of approximately 30 seconds per grinding are usually sufficient. Zirconia (ZrO_2) abrasive papers will last longer than silicon carbide or Al_2O_3 under these grinding conditions. The loaded sample holder is then ultrasonically cleaned in alcohol, dried in an air blast and returned to the machine for polishing.

Two or three polishings may be employed. One approach, which applies primarily to holders containing up to six 30 mm (1.25 inch) diameter mounts, begins with $6\mu m$ diamond on a hard, napless chemotextile, proceeds to $3\mu m$ diamond on a low-nap cloth and finishes with $1\mu m$ diamond on a soft, high-nap cloth. Wheel speed in each case is 150 rpm. The first and second polishings use a pressure of 150 N (35 lbf) for 2 min per step; the final polishing, 100 N (25 lbf) pressure for 35 seconds. Polishing extender is dripped sparingly on the wheels during each step, and the sample holder and work wheels should rotate in the same direction in each step.

Another approach, useful for high volume production of embedded and unembedded samples, is to use a 9μm diamond slurry on a lapping disk, followed by a 0.3μm Al_2O_3 suspension on a low-nap rayon cloth, with an optional intermediate step of 3μm Al_2O_3 suspension on a hard chemotextile. Times and pressures are varied to suit the size and number of samples.

The sample holder is ultrasonically cleaned and dried after each polishing, and the samples are then removed from the holder for etching and final examination. Specimens of different alloys and hardnesses usually may be mixed in a single holder without harming the prepared surfaces of the softer samples.

Electropolishing. Clean, as-rolled strip surfaces or sectioned sample faces of beryllium copper alloys prepared through 400 to 600 grit grinding paper may be electropolished. A satisfactory all-purpose electrolyte for beryllium copper alloys is a mixture of 1 part nitric acid (HNO_3) and 2 parts methanol used at a temperature of -30°C (-20°F), with a voltage of 25 V and a platinum cathode. An electrolyte of 40 ml phosphoric acid (H_3PO_4), 60 ml hydrogen peroxide (H_2O_2), 40 ml methanol, and 20 ml H_2O may also be used for beryllium copper in conjunction with a stainless steel cathode, a mask of 0.5 to 1 cm^2 (0.08 to 0.16 in^2) area, 20 to 30 V, and approximately 0.2 A. This technique is applicable to general polishing, but is particularly suited to examining intermetallic phases in beryllium copper, which can be rendered in high relief. Polishing is accomplished in a few seconds to a few minutes, using a moderate electrolyte velocity.

Macroexamination

Castings, forgings, billet, hot-rolled plate and hot-extruded rod, bar, and tube forms of beryllium-containing alloys are frequently subjected to low magnification macroexamination. One purpose of macroexamination is to evaluate grain structure and metal flow patterns indicating thermo-mechanical processing history. The technique also applies to documentation of differential heat treatment, weld penetration and localized structural damage due to environmental attack.

Macroetching. Once ground to at least 320 or 400 grit, beryllium copper is macroetched by an initial, brief immersion in concentrated HNO_3, followed by immersion in or flooding with dilute HNO_3 (1 part concentrated HNO_3 to 2 parts H_2O). The etchant attack is stopped by rinsing in running water. After the etched sample is rinsed in alcohol and dried in a warm air blast, the macrostructure can be preserved by spraying on a coat of clear lacquer, preferably containing a copper tarnish inhibitor such as benzotriazole ($C_6H_4NHN{:}N$).

Beryllium nickel can be macroetched by the same dilute HNO_3 procedure as for beryllium copper. The concentrated HNO_3 presoak is omitted to avoid pitting.

In addition to grain structure and flow patterns, macroetched samples of beryllium coppers with 1.6 wt% or more Be reveal locally heat-affected zones as light etched areas if reannealed or unaged and as dark etched areas if aged. Light etched or reddish colored areas adjacent to exterior or crack surfaces in uniformly aged materials usually signal environmental attack leading to local depletion of beryllium and lack of aging response. Matrix darkening on etching is not as pronounced in aged beryllium nickel and is absent in beryllium coppers containing less than 0.6 wt% Be, limiting the information revealed by macroetching of these alloys essentially to matters of grain morphology. Castings and cast billet exhibit columnar dendritic and/or equiaxed grain growth depending upon solidification conditions. Hot-finished, large section forms of the beryllium-containing alloys occasionally exhibit in the macrostructure individual large grains, elongated in the direction of working.

Microexamination

Etching procedures for beryllium-containing alloys vary with alloy type and condition or temper. Particularly for beryllium coppers with less than 0.6 wt% Be, general microstructures are more difficult to reveal in the age hardened conditions than in the hot-finished, solution annealed or cold worked conditions.

Etchants for beryllium copper and beryllium nickel are listed in Table 1, along with their compositions, etching procedures, uses and precautions. Nitric acid and water (Etchant 9, Table 1) is a good, general purpose etchant for all forms and tempers of beryllium nickel. Modified Marble's etchant (Etchant 10, Table 1), which is especially suited to hot worked or annealed material, provides dramatic grain structure detail with sensitive tint illumination.

Ammonium persulfate/ammonium hydroxide (Etchant 1, Table 1) is a general purpose etchant for beryllium coppers. It reveals grain structure in unaged material, although twinning may be present to complicate grain size measurement. In the case of alloys with 1.6 to 2.75 wt% Be, this etchant darkens the matrix of age hardening material; the degree of coloration varies with the extent of precipitation in the alloy. A variation of this etchant that contains H_2O_2 (Etchant 2, Table 1) offers improved grain boundary delineation in unaged material.

Grains in alloys with higher beryllium content may be highlighted by etching with ammonium persulfate/ammonium hydroxide, followed by brief swabbing with dichromate (Etchant 3, Table 1) to lighten the matrix. Another reliable way to enhance grain boundaries in unaged alloys of these compositions is to age the samples for 15 to 20 minutes at 370°C (700°F), then follow with the two-stage etching procedure described above. This technique eliminates twinning, and grain size can be accurately determined from the decoration of the grain boundaries with dark etching gamma precipitate.

Alloys with 0.6 wt% or less Be exhibit little microstructural difference between the aged and unaged conditions, and grain structure is frequently obscured by twinning if ammonium persulfate/ammonium hydroxide is used. To enhance the general microstructure in these situations, the cyanide (Etchant 6, Table 1), persulfate hydroxide/cyanide (Etchant 7, Table 1), or two-step cyanide peroxide hydroxide (Etchant 8, Table 1) etchants are used. Care must be taken to observe all safety precautions of these toxic solutions.

None of the etchants listed differentially attack intermetallic compounds in beryllium copper — to distinguish cobalt or nickel beryllides from beta phase, for example. This distinction must be made on the basis of appearance in the as-polished condition. Beryllides are blue-gray; beta phase is creamy white and surrounded by a thin, dark outline.

Any of the etchants listed, which are intended for bright-field optical microscopy, may be utilized for scanning electron microscopy examination to reveal fine structural details not optically resolvable. One such application is the resolution of lamellar structure of grain-boundary gamma precipitate in high temperature aged beryllium coppers containing 1.60 to 2.00 wt% Be.

Microstructures of Beryllium Copper Alloys

The two general categories of beryllium copper alloys are the high strength and the high conductivity alloys. The wrought high strength alloys contain 1.60 to 2.00 wt% Be, with approximately 0.25 wt% Co. The addition of cobalt promotes fine grain size in the cast form, lessens grain growth during annealing and reduces the rapid softening of the alloy due to overaging. Solution annealing at temperatures of 760 to 790°C (1400 to 1450°F), followed by rapid quenching, retains the beryllium in solid solution at room temperature. Precipitation hardening can be accomplished by aging for 0.1 to 4 hr at 260 to 400°C (500 to 750°F); the time and temperature depend on the composition, amount of cold work and strength levels desired. Cold working prior to aging results in faster age hardening and higher strengths.

The most commercially important of the high strength compositions is C17200, which contains 1.80 to 2.00 wt% Be. This is the strongest of the wrought beryllium copper alloys; tensile strengths range to 1520 MPa (220 ksi) in the cold worked and fully age hardened condition. A leaded version of this alloy, C17300, exhibits improved machinability. A composition slightly lower in cost, C17000, with 1.60 to 1.79 wt% Be, is available with tensile properties in the age hardened condition approximately 10% lower than those of C17200.

The high strength alloys are also produced as casting alloys, designated C82400, C82500, C82510, C82600, and C82800. The beryllium content is higher (up to approximately 2.75 wt%) than in wrought alloys, but in general microstructural characteristics are similar. These alloys are produced as cast ingots that can be remelted and cast by foundries using any conventional molding technique.

The high conductivity alloys have low beryllium levels (0.2 to 0.7 wt%) and high cobalt or nickel levels. The wrought version of the alloy containing 2.4 to 2.7 wt% Co is designated C17500. The cast version is designated C82000. The wrought version containing 1.4 to 2.2 wt% Ni instead of cobalt is designated C17510, and the corresponding casting alloy is C82200. The wrought version of the alloy containing 0.35 - 0.60 wt% Co is designated C17410. The properties of the nickel-containing alloys are very similar to the cobalt-containing alloys. The solution annealing temperature range for the high conductivity alloys is 900 to 955°C (1650 to 1750°F). Aging is performed at 425 to 565°C (800 to 1050°F) for 3 to 8 hr, depending on the amount of cold work and combination of properties sought.

The various commercial beryllium copper alloys illustrated in this article are listed in Table 2, which provides compositional limits. Data sheets published by the alloy producers may be consulted for detailed information, such as physical and mechanical properties and typical applications. The phases and constituents resulting from alloying elements and various heat treatments are discussed below.

The Beryllide Phase. Commercial beryllium copper alloys contain cobalt or nickel or both. These alloying elements are normally in solution in the liquid metal. Because of their strong affinity for beryllium, they combine with it and separate during solidification as particles that are approximately 10μm in the largest dimension. These constituent particles are termed "beryllides." During subsequent thermomechanical processing, the beryllides are broken up somewhat but are not appreciably dissolved into solid solution during normal solution annealing.

The primary beryllide phase is best observed in the as-polished condition as blue-gray Chinese script in castings. The secondary beryllides forming after solidification of the major phase can have a rod-like morphology with preferred crystallographic orientation with the matrix. In wrought products, the beryllides appear as roughly spherical blue-gray particles.

The Beta Phase. The β phase forms peritectically from the liquid metal. It is observed in high strength alloy castings as an interdendritic network surrounding the primary copper-rich alpha phase. Experiments have shown that the phase cannot be retained at room temperature, because it decomposes into alpha and β phases by a eutectoid transformation. The (transformed) β phase stands out in relief in the as-polished state as white angular patches. In wrought strip containing 1.8 to 2.0 wt% Be, long (transformed) β stringers are seldom found, but in heavier section products, plate, tubing or rod, some β phase will be found. Homogenization before hot working, followed by cold working and annealing tends to reduce the amount of β phase.

The Gamma Phase. The γ phase forms in overaged beryllium copper alloys as an equilibrium precipitate. In the alloys with high beryllium content, the γ phase precipitation starts at the grain boundaries and advances into the adjoining grains, consuming the fine, metastable precipitates. This type of precipitation is termed "discontinuous precipitation" or "cellular precipitation." The γ precipitates formed by this mechanism have a platelike morphology. In the age hardened state, the grain boundary containing the γ precipitate is soft compared to the hardened matrix.

Precipitation of the γ phase can also occur in the grain boundaries in high strength beryllium copper alloys if the rate of quenching from the solution annealing temperature is not fast enough to retain beryllium in solid solution. In metallographically polished specimens etched using standard procedures, the γ phase stands out at the grain boundaries as dark nodules on a bright matrix. The lamellar morphology of the γ phase is resolved by scanning or transmission electron microscopy.

Hardening Precipitates. Excellent room temperature mechanical properties of beryllium copper alloys are derived from the formation of a series of metastable precipitates during aging. Several metastable phases form before the equilibrium γ phase is observed. The precipitation sequence of these alloys occurs by the following sequence after annealing, quenching and aging at a relatively low aging temperature:

Supersaturated solid solution $\rightarrow$
Guinier-Preston (GP) zones $\rightarrow \gamma'' \rightarrow \gamma' \rightarrow \gamma$

Guinier-Preston zones are the first precipitates to form and are coherent with the matrix. They are nucleated in large densities. The coherency strain fields set up from the misfit of the GP zones and the matrix strengthen the alloy. With continued aging, Guinier-Preston zones transform to other metastable precipitates.

The metastable precipitates can be detected by transmission electron microscopy. They are identified by the characteristic streaks and extra reflections observed in the electron diffraction pattern. The presence of hardening precipitates can be recognized only indirectly by light microscopy as striations on the surface of a polished and etched alloy that result from the overlap of coherency strains.

Microstructures of Beryllium Nickel Alloys

Commercial beryllium nickel alloys can be precipitation hardened in a manner similar to beryllium copper alloys by solution annealing, quenching, optional cold working, and aging. These alloys usually contain 1.80 to 2.70 wt% Be. Titanium, chromium, or carbon, among others, are added for grain size control, corrosion resistance, hot workability, or machinability. Typical chemical compositions of the alloys are listed in Table 3. The solution annealing temperature for these alloys is performed at 980 to 1065°C (1800 to 1950°F). After quenching, tensile strengths approaching 2068 MPa (300 ksi) are obtained by aging at 510°C (950°F) for 1 to 3 hr.

Precipitation in the beryllium nickel system is similar to the beryllium copper system regarding the sequence and the structure of the metastable phases formed during aging. The equilibrium phase (NiBe) is formed by a discontinuous reaction consuming the metastable hardening precipitates. Nickel beryllium compound particles containing titanium exist in the wrought alloy and assist grain refinement. Graphite nodules are present in the cast alloys containing carbon and contribute to improved machinability.

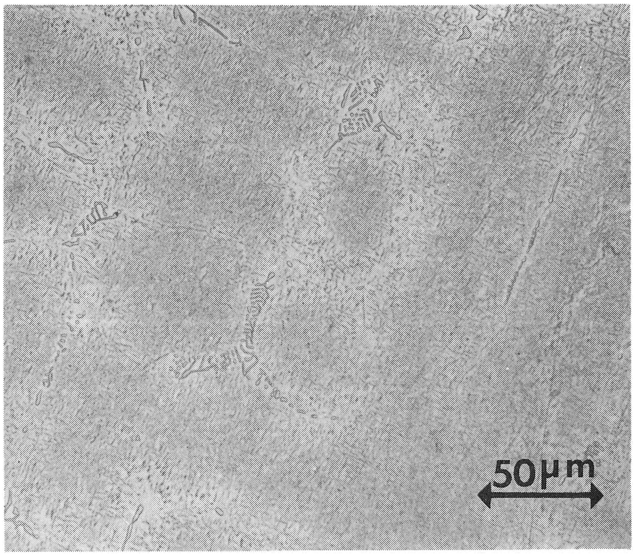

Figure 1 - C82200 alloy casting. As-cast microstructure shows interdentritic network of large primary beryllide phase in a matrix of alpha solid solution. Preferred orientation of small secondary beryllides is observed within the matrix. (Etchant 6, Table 1).

Figure 2 - C82500 alloy casting, solution annealed at 790°C (1450°F) and aged to peak hardness at 315°C (600°F) for 3 hr. Microstructure consists of Chinese script beryllides in a copper-rich solution matrix, with angular beta phase transformed to a lamellar aggregate of alpha and gamma phase. Striations are the result of metastable precipitation in the alloy. (Etchant 1, Table 1).

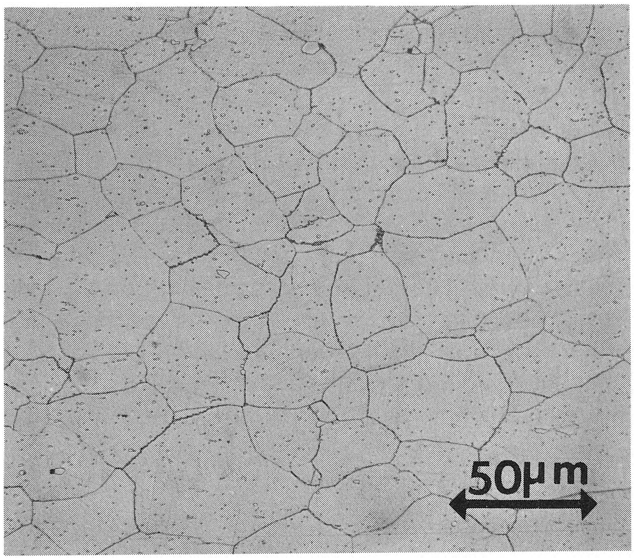

Figure 3 - C17200 alloy strip, mill hardened to AM (TM00) temper to achieve maximum formability at moderate strength. Longitudinal section shows roughly equiaxed grains of alpha phase with smaller cobalt beryllide particles. Metastable precipitates that form during hardening and increase strength and hardness cannot be resolved optically. (Etchant 1, Table 1).

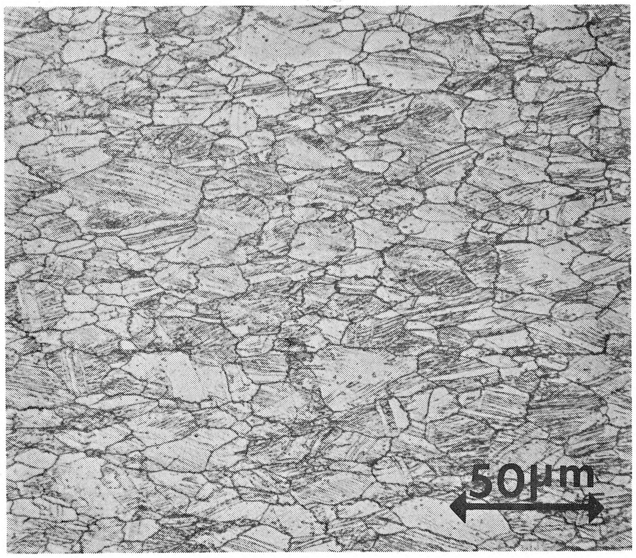

Figure 4 - C17200 alloy strip, mill hardened to XHMS (TM08) temper for high strength and limited formability. Longitudinal section shows elongated grains of alpha phase and cobalt beryllides. Striations result from precipitation of metastable phases not resolved by optical microscopy. (Etchant 1, Table 1).

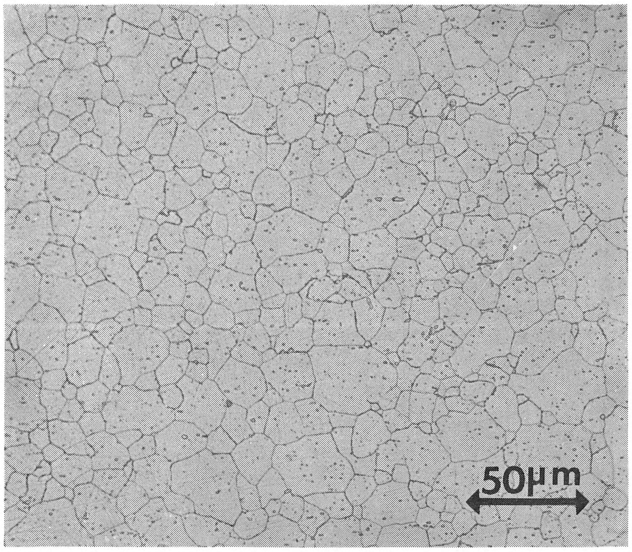

Figure 5 - C17200 alloy strip, solution annealed at 790°C (1450°F) and water quenched. Longitudinal section shows equiaxed grains of supersaturated alpha phase solid solution of beryllium in copper. Cobalt beryllide particles are uniformly dispersed throughout the structure. (Etchant 1 or 2, Table 1).

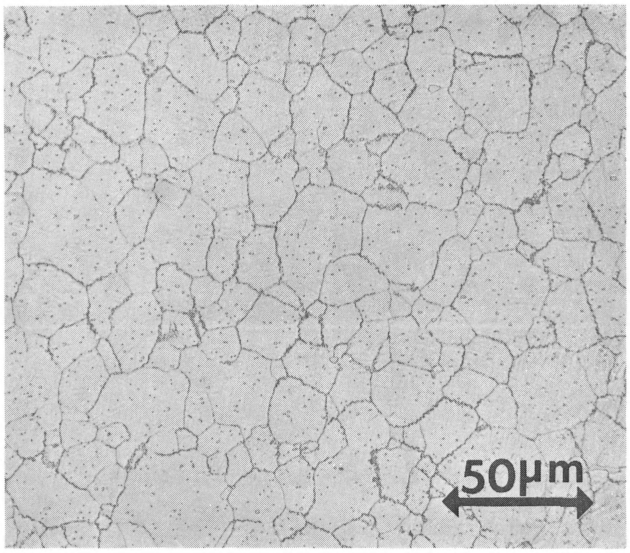

Figure 6 - C17200 alloy strip, solution annealed at 790°C (1450°F), quenched rapidly to room temperature and precipitation hardened at 315°C (600°F) for 3 hr to achieve maximum hardness. Longitudinal section shows equiaxed alpha grains with small cobalt beryllide particles. Metastable phases are not resolved, but small quantities of equilibrium gamma phase are present in the grain boundaries. (Etchant 1, Table 1).

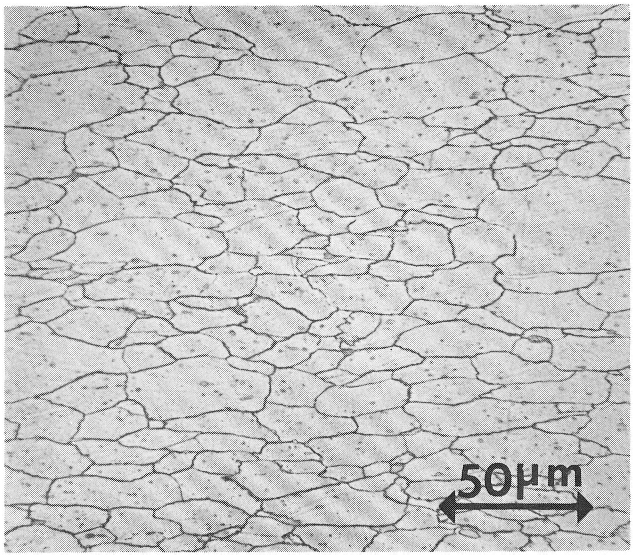

Figure 7 - C17200 alloy strip, solution annealed at 790° (1450°F) and cold rolled 37% to full hard temper. Longitudinal sections shows elongated grains of alpha phase and cobalt beryllides. (Etchant 1 or 2, Table 1).

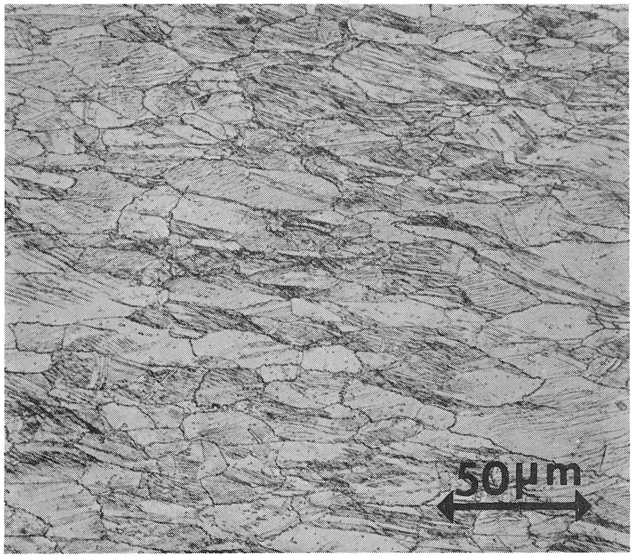

Figure 8 - C17200 alloy strip, solution annealed, cold rolled full hard, and precipitation hardened at 315°C (600°F) for 2 hr to achieve hardness. Longitudinal section shows elongated grains of alpha phase and cobalt beryllides. Striations are caused by metastable precipitates not resolved by optical microscopy. (Etchant 1, Table 1).

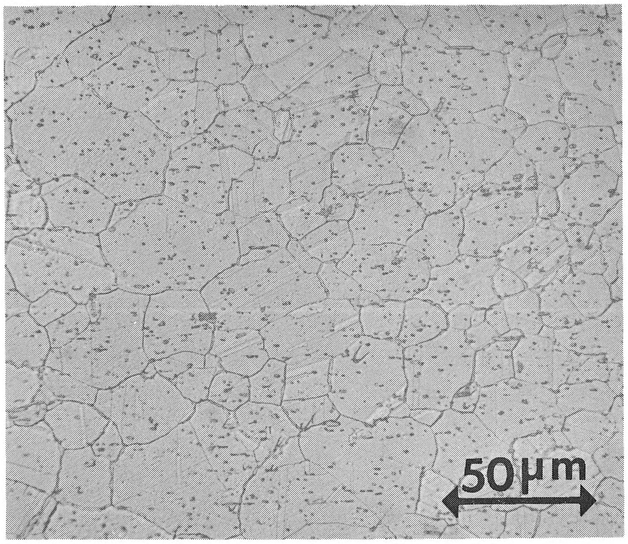

Figure 9 - C17510 alloy strip, solution annealed at 900°C (1650°F), quenched rapidly to room temperature, and precipitation hardened at 480°C (900°F) for 3 hr to achieve maximum hardness. Equiaxed grains of alpha supersaturated solution of beryllium and small nickel beryllide particles are shown. (Etchant 6, Table 1).

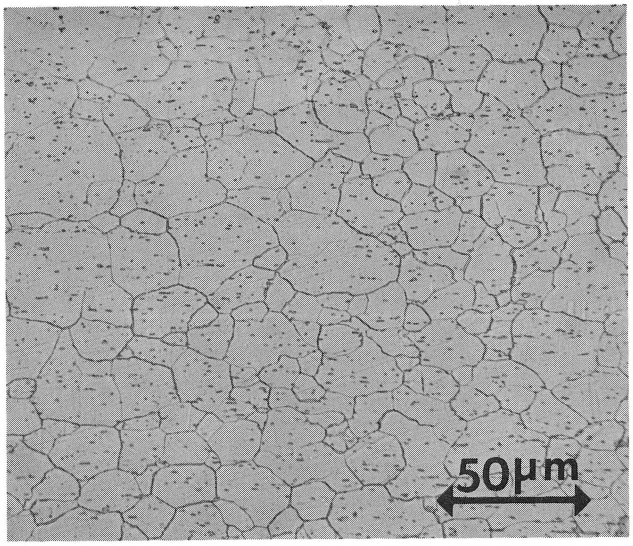

Figure 10 - C17510 alloy strip, solution annealed, cold rolled, and precipitation hardened at 480°C (900°F) for 2 hr to achieve maximum hardness. Structure consists of alpha phase and a uniform distribution of small nickel beryllide phase. Elongated grains are the result of cold work. Metastable precipitates are not resolved. Etchant 6, followed by a swabbing with Etchant 8 (Table 1).

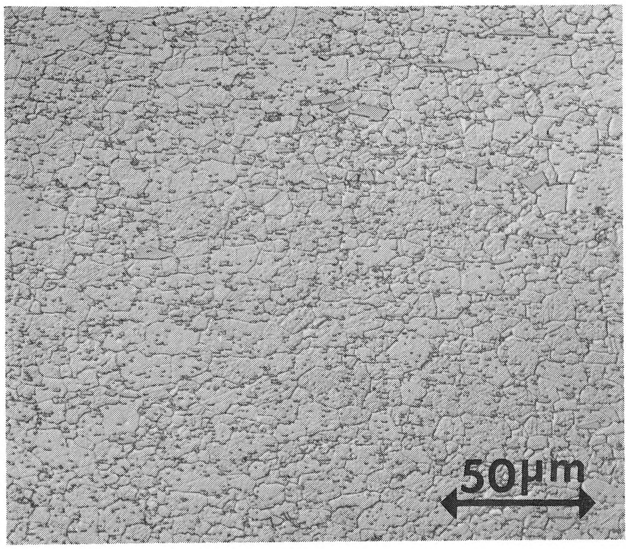

Figure 11 - C17500 alloy strip, solution annealed, and precipitation hardened at 480°C (900°F) for 3 hr to achieve maximum hardness. Microstructure shows equiaxed grains of alpha supersaturated solution of beryllium and cobalt in copper. Small cobalt beryllide particles are uniformly distributed, and metastable hardening precipitates are not resolved. (Etchant 6, Table 1).

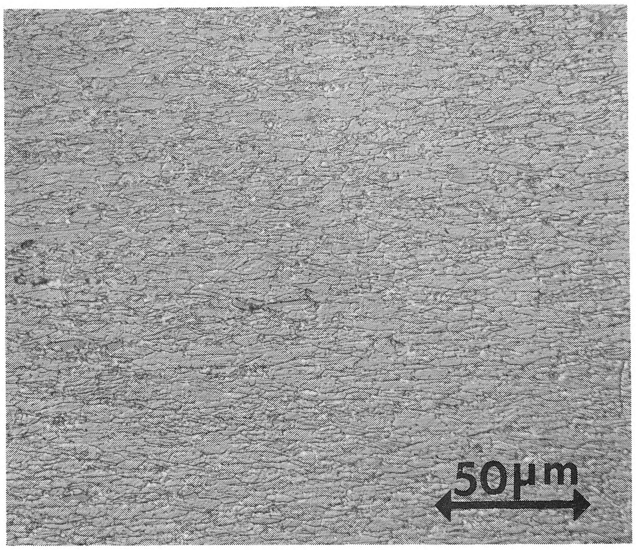

Figure 12 - C17500 alloy strip, solution annealed, cold rolled, and precipitation hardened at 480°C (900°F) for 2 hr to achieve maximum hardness. Structure consists of alpha phase and a uniform distribution of small cobalt beryllide particles. Elongated grains are the result of cold work, and metastable hardening precipitates are not resolved. (Etchant 6, Table 1).

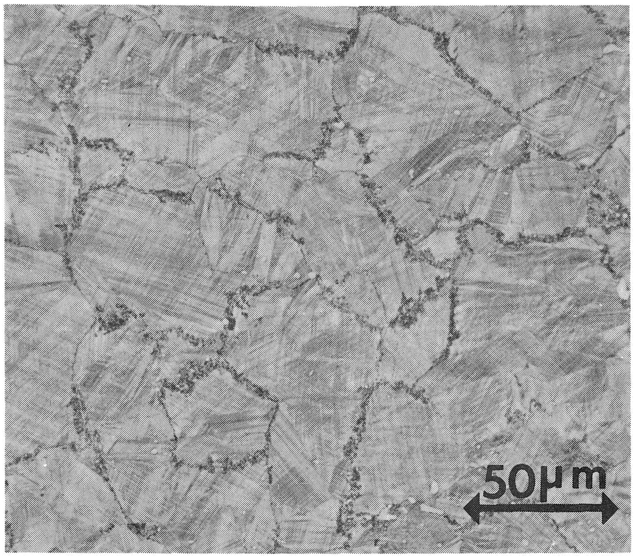

Figure 13 - C17200 alloy strip, solution annealed and aged at 370°C (700°F) for 6 hr to attain an overaged condition. The structure shows gamma precipitates in the grain boundaries, which appear as dark nodules in a light matrix. (Etchant 1, Table 1).

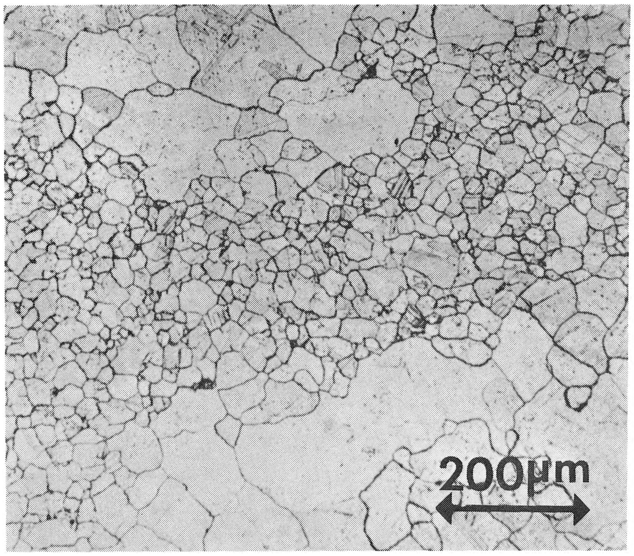

Figure 14 - C17200 alloy plate, cast, homogenized, and hot worked. The microstructure shows nonuniform distribution of grain sizes, which is typical of a hot worked product. Greater uniformity in grain size distribution may be achieved in the finished product by successive cold working and annealing operations. (Etchant 1, Table 1).

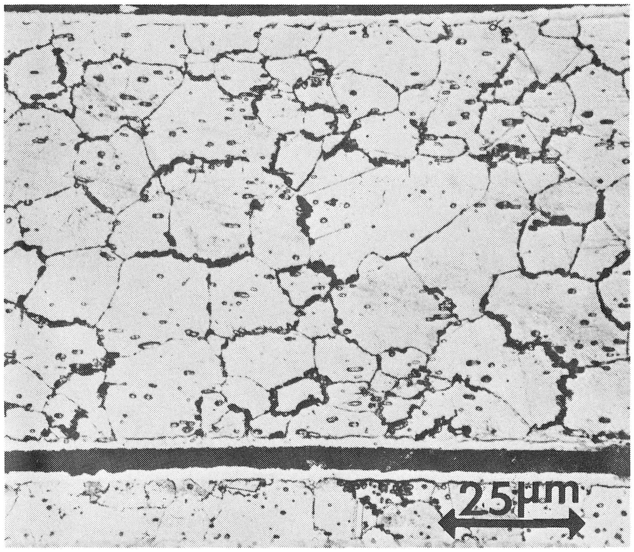

Figure 15 - N03360 alloy strip, solution annealed at 990°C (1800°F) water quenched, and aged at 510°C (950°F) for 1.5 hr. The structure shows nickel beryllium compound particles dispersed uniformly through the nickel-rich matrix. Hardening precipitates are not resolved, but equilibrium gamma (NiBe) is present in grain boundaries. (Etchant 10, Table 1).

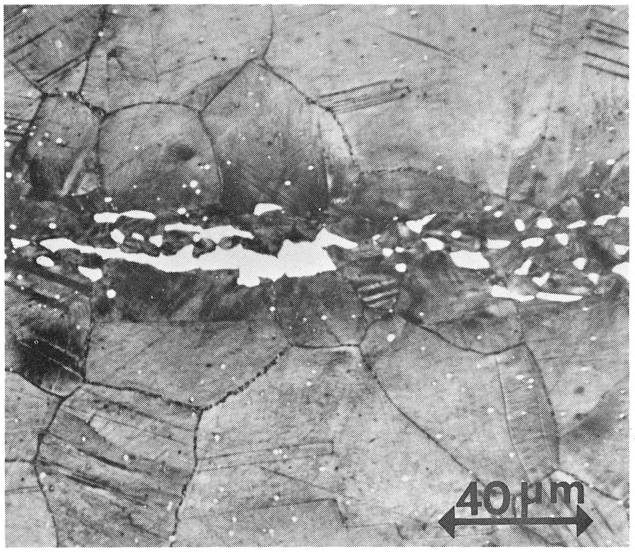

Figure 16 - C17200 alloy strip, solution annealed, and age hardened. The white constituents of the structure are beta stringers. These zones of beryllium segregation are from billet casting. (Etchant 1, Table 1).

Figure 17 - C17200 alloy strip heated to 885°C (1625°F) and water quenched. The microstructure shows "burned metal" caused by solution annealing at too high a temperature. Incipient melting at the grain boundaries, caused by extreme temperature, resolidifies as beta phase. (Etchant 1, Table 1).

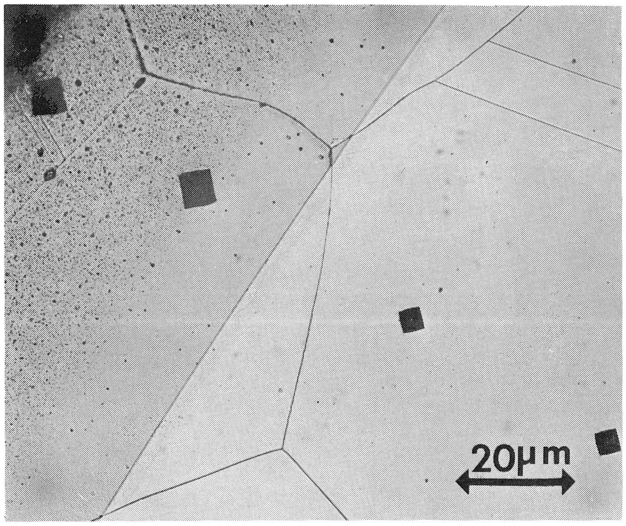

Figure 18 - C17510 alloy rod, solution annealed in air at 980°C (1800°F) for 3 hr, then aged at 480 °C (900°F) for 3 hr. The microstructure shows internal oxidation resulting from solution annealing without a protective atmosphere. Note the loss in hardness (as indicated by the microhardness indentations) within the internal oxidation zone. (Etchant 6, Table 1).

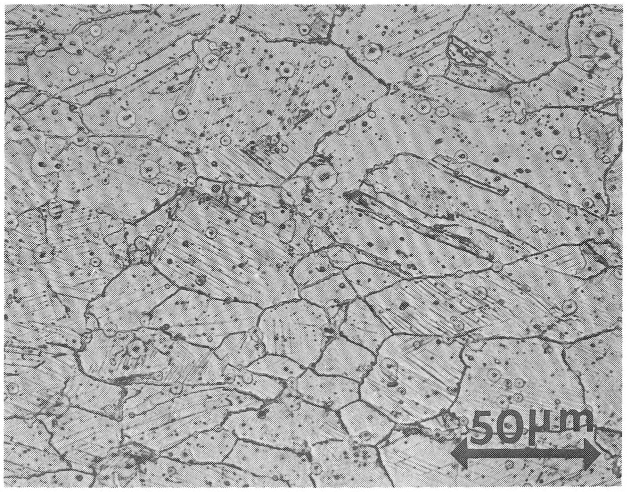

Figure 19 - Lead-containing C17300 rod, solution annealed, quenched rapidly to room temperature, and precipitation hardened at 315°C (600°F.) for 3 hr, to achieve maximum hardness. Cobalt beryllide particles similar to C17200 are shown. In addition, the microstructure shows uniform distribution of lead particles, appearing as fine particles inside circles which are etching artifacts. The presence of lead particles is made visible through a special etching process, consisting of swab etching for 2 to 3 seconds in a potassium dichromate-sulfuric acid-hydrochloric acid solution (Etchant 3, Table 1), and rinsing, followed by etching again by emersion in a 1% potassium cyanide solution in water. (Etchant 6, Table 1).

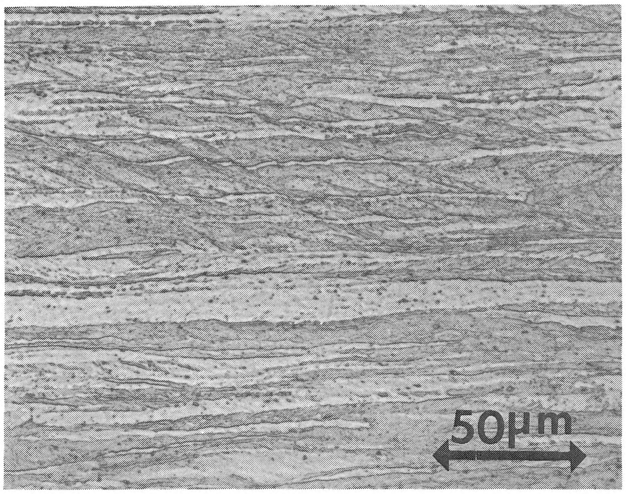

Figure 20 - C17410 strip, mill hardened to HT temper to achieve high strength, high conductivity and superior stress relaxation resistance. Longitudinal section shows elongated grain structure. Metastable precipitates are not resolved by optical microscopy.

Table 1: Etching reagents for beryllium copper and beryllium nickel alloys

Etchant	Composition	Comments
1. Ammonium persulfate/ ammonium hydroxide	1 part NH_4OH (conc) and 2 parts $(NH_4)_2S_2O_8$ (ammonium persulfate) 2.5% in H_2O (a)	All beryllium copper alloys. General structure. Preheat sample in hot water (optional); swab etch 2-20 seconds; use fresh.
2. Ammonium persulfate/ ammonium hydroxide (variation)	2 parts 10% $(NH_4)_2S_2O_8$, 3 parts NH_4OH (conc), 1 part 3% H_2O_2, and 5-7 parts H_2O (a)	All beryllium copper alloys. Offers improved grain boundary delineation in unaged material. A, ¼H, ½H, H tempers (unaged) use less H_2O. AT through HT (aged) use more H_2O. Use fresh; swab or immerse 5-60 seconds. Preheat specimen in hot H_2O if etching rate is slow.
3. Dichromate	2 g $K_2Cr_2O_7$ (potassium dichromate), 8 ml, H_2SO_4 (conc), 1 drop HCl per 25 ml of solution, and 100 ml H_2O (a)	Grain structure of wrought C17000, C17200, C17300. Use for AT through HT and mill hardened (aged). Etch first with ammonium persulfate hydroxide (No. 1 or 2); wipe dichromate 1-2 times over specimen to remove dark etch color. Do not overetch; sample may pit. May be used with laboratory aging of annealed or as-rolled material at 370°C (700°F) for 15 to 20 minutes to enhance grain boundary delineation for grain size determination.
4. Hydroxide/peroxide	5 parts NH_4OH (conc), 2-5 parts 3% H_2O_2, and 5 parts H_2O (a)	Common etchant for copper and brass, also applicable to beryllium copper alloys. Use fresh.
5. Ferric chloride	5 g $FeCl_3$ (ferric chloride), 50 ml HCl, and 100 ml H_2O (a)	Common etchant for copper alloys, also applicable to cold rolled tempers of beryllium copper alloys C17500 and C17510 to show grain structure. Immerse 3-12 seconds.
6. Cyanide	1 g KCN (potassium cyanide) and 100 ml H_2O (a)	General structure of beryllium copper alloys C17500, C17510 (No. 6). Immerse 1.5 minutes; stir slowly while etching; use Etchant 7 if others are too weak to bring out structure. A two-step technique for improved results on C17510 includes immersion in Etchant 6 followed by swabbing with Etchant 8. **Caution:** Poison fumes! Use fume hood. Do not dispose of used solutions directly into drains. Pour used solution into breaker containing chlorine bleach. Let stand 1 hour to neutralize.
7. Persulfate hydroxide/ cyanide	4 parts ammonium persulfate hydroxide etchant (Etchant 1 or 2) and 1 part cyanide etchant (Etchant 6)	
8. Cyanide peroxide hydroxide	20 ml KCN, 5 ml H_2O_2, and 1-2 ml NH_4OH	
9. Nitric acid and water	30 ml HNO_3 (conc) and 70 ml H_2O (a)	Beryllium nickel, all tempers. General structure. Swab etch.
10. Modified Marble's etchant	4 g $CuSO_4$ (copper sulfate), 20 ml HCl (conc), and 20 ml H_2O (a)	Beryllium nickel, all tempers. General structure. Swab etch. May also be used with sensitive tint illumination to reveal grain structure of hot worked or annealed material.
11. Phosphoric acid electrolyte	20 ml H_2O (tap, not distilled), 58 ml 3% H_2O_2, 48 ml H_3PO_4, and 48 ml ethyl alcohol	For deep etching of beryllium copper. Polish specimen through 1μm or finer Al_2O_3. Use 0.5-1cm^2 (0.08-0.16 in^2) mask. Use 0.1 A to etch (higher amperes to polish), low to moderate flow rate; 3 to 6 seconds to etch, up to 60 seconds to polish.

(a) Use distilled water, unless otherwise noted.

Table 2: Chemical compositions of beryllium copper alloys

Brush Alloy	UNS #	Composition, wt%					
		Be	Co	Ni	Pb	Other	Cu
Wrought Alloys							
165	C17000	1.60-1.79	(a)	(a)	—	—	rem
25	C17200	1.80-2.00	(a)	(a)	—	—	rem
M25	C17300	1.80-2.00	(a)	(a)	0.20-0.6	—	rem
174	C17410	0.15-0.50	0.35-0.60	—	—	—	rem
3	C17500	0.40-0.7	2.4-2.7	—	—	—	rem
10	C17510	0.20-0.6	0.30 (b)	1.4-2.2	—	—	rem
Cast Alloys							
10C	C82000	0.45-0.8	2.4-2.7	0.20	—	0.15 Si	rem
3C	C82200	0.35-0.8	—	1.0-2.0	—	—	rem
165C, 165CT	C82400	1.65-1.75	0.20-0.40	0.10	—	0.20 Fe	rem
20C, 20CT	C82500	1.90-2.15	0.35-0.7	0.20	—	0.20-0.35 Si, 0.20 Fe	rem
21C	C82510	1.90-2.5	1.0-1.2	0.20	—	0.20-0.35 Si, 0.20 Fe	rem
245C, 245CT	C82600	2.25-2.45	0.35-0.7	0.20	—	0.20-0.35 Si, 0.25 Fe	rem
275C, 275CT	C82800	2.50-2.75	0.35-0.7	0.20	—	0.20-0.35 Si, 0.25 Fe	rem

(a) Nickel + Cobalt, 0.20 min; Nickel + Cobalt + Iron, 0.60 max.
(b) Maximum, if no range given

Table 3: Chemical compositions of nickel alloys

Alloy	Type	Composition, wt%				
		Be	Ti	Cr	C	Ni
UNS N03360	Wrought	1.80-2.05	0.4-0.6	—	—	rem
220C	Casting	2.00-2.30	—	—	0.50-0.75	rem
41C	Casting	2.7	—	0.5	—	rem
42C	Casting	2.7	—	12	—	rem
43C	Casting	2.7	—	8	—	rem

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