Metallographic Specimen Preparation for Electron Backscattered Diffraction

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Electron backscattered diffraction (EBSD) is performed with the scanning electron microscope (SEM) to provide a wide range of analytical data; e.g., crystallographic orientation studies, phase identification and grain size measurements. The quality of the diffraction pattern, which influences the confidence of the indexing of the diffraction pattern, depends upon removal of damage in the lattice due to specimen preparation. It has been claimed that removal of this damage can only be obtained using electrolytic polishing or ion-beam polishing. However, the use of modern mechanical preparation methods, equipment and consumables does yield excellent quality diffraction patterns. The experiments discussed here covered a wide variety of metals and alloys prepared mechanically using three to five steps, based on straightforward methods that generally require less than about twenty-five minutes.

KEYWORDS:
mechanical specimen preparation, diffraction patterns, deformation, relief control, flatness

INTRODUCTION

Electron backscattered diffraction (EBSD) is performed with the scanning electron microscope (SEM) to provide a wide range of analytical data; e.g., crystallographic orientation studies, phase identification and grain size measurements. A diffraction pattern can be obtained in less than a second, but image quality is improved by utilizing a longer scan time. Grain mapping requires development of diffraction patterns at each pixel in the field and is a slower process. The quality of the diffraction pattern, which influences the confidence of the indexing of the diffraction pattern, depends upon removal of damage in the lattice due to specimen preparation. It has been claimed that removal of this damage can only be obtained using electrolytic polishing or ion-beam polishing. However, the use of modern mechanical preparation methods, equipment and consumables does yield excellent quality diffraction patterns without use of dangerous electrolytes and the problems and limitations associated with electropolishing and ion-beam polishing. Basically, if mechanical preparation results in quality polarized light images of non-cubic crystal structure elements and alloys (e.g., Sb, Be, Hf, α-Ti, Zn, Zr), or color tint etching of cubic, or non-cubic crystal structure elements or alloys produces high-quality color images, then the surface is free of harmful residual preparation damage and EBSD patterns with high pattern quality indexes will be obtained. Because of the acute angle between the specimen and the electron beam (70 – 74°), exceptional surface flatness is also necessary for best results.

Polarized light image quality is dependent upon the elimination of preparation damage and upon the quality of the microscope optics [1]. Consequently, always check the polarized light response of metals that will respond to polarized light, to verify preparation quality before performing EBSD. For cubic metals, etch first with a general-purpose reagent to confirm the nature of the expected microstructure. Then, repeat the final polishing step and use a color tint etch [1,2] to verify freedom from damage. EBSD is best performed with an as-polished, non-etched specimen due to the steep angle to the electron beam, as surface roughness can degrade the diffraction pattern. A well-prepared, un-etched specimen will exhibit a good grain-contrast image with a backscattered electron detector [3]; another good test for freedom from surface damage.

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DEVELOPMENT OF PREPARATION METHODS

Specimen preparation methods for metals and alloys have been developed [4] that yield excellent results using straightforward methods that generally require less than about twenty-five minutes. High-purity metals require more preparation time than alloys. Automated preparation equipment is recommended, as the methods will be performed accurately and reproducibly. Manual ("hand") preparation cannot produce flatness, phase retention and damage removal as easily as automated processing and is less reproducible.

Successful preparation requires that sectioning be performed with equipment and consumables that minimize damage. Sectioning is a violent process and it can introduce massive damage. Crystal structure does influence damage depth; face-centered cubic metals exhibit greater damage than body-centered cubic metals for the same preparation procedure because fcc metals slip more readily than bcc metals. Use only abrasive blades designed for metallography that are recommended for the specific metal/alloy in question. A precision saw yields even less damage as the blades are much thinner and the applied loads are much lower. Cutting with machines and blades/wheels that introduce minimal damage is the most critical step in generating damage-free metallographic surfaces; this cannot be over-emphasized. Then, commence grinding with the finest possible abrasive and surface that will make all of the specimens in the holder co-planar and remove the sectioning damage in reasonable time. This is the second critical rule for obtaining damage-free polished surfaces. The proposed methods utilize flat, woven cloths or pads that minimize relief problems. To minimize damage, use less aggressive surfaces, such as silk, nylon, polyester or polyurethane. The specimen preparation method must remove all scratches. If scratches are present, so to is damage below the scratch. Scratch depths produced in grinding and polishing are not uniform. A deep scratch will have deep deformation below it. The preparation method must remove the scratches and the underlying da-

mage in order to obtain high quality EBSD patterns.
The experiments discussed here covered a wide variety of metals and alloys prepared mechanically using three to five steps. The EBSD patterns shown were developed using both the EDAX-TSL and Oxford Instruments HKL systems on a variety of scanning electron microscopes (SEM) using tungsten, LaB_6 and field emission electron sources. The plane-of-polish was oriented between 70 and 74° from horizontal, depending upon the system used. The TSL system generates pattern quality indexes, PQI, and the results shown here are the average and 95% confidence limits for 25 randomly selected grains using unetched specimens. The high-purity metallic samples were analyzed using the HKL Channel 5 EBSD system. These patterns were evaluated using the band contrast data, with the average and standard deviation calculated for a number of measurements. Several cast specimens had very large grains, so only a few EBSD patterns could be obtained. The silicon specimen was a single crystal so all patterns were basically identical.

RESULTS

The first examples presented will be a wrought, cold worked, high-purity (99.999%) aluminum and an Al – 7.12 % Si casting alloy. Al is a difficult EBSD subject as the low atomic number is inefficient in generating backscattered electrons. High-purity metals are always far more difficult to prepare than commercial-purity metals while alloys are the easiest to prepare. EBSD patterns will be more difficult to generate on a wrought, non-recrystallized, cold worked specimen due to the resulting distortion of the crystal lattice. So, combining both the high-purity and non-recrystallized conditions makes for an extreme test of the preparation method. The table below presents the test method used, except that the specimen in this case was not subjected to a vibratory polish after use of the five-step preparation method. The band contrast value averaged 151.1 after using the five-step method. It is our experience, as discussed below that using a 20-minute vibratory polish after the standard preparation cycle will improve the band contrast at least 10%. Longer times will yield further improvements. When developing grain maps, maximizing the band contrast, or the pattern quality index, produces greater confidence in indexing; this is vital when indexing several hundred points per second.
Shown below in Figure 1 is the cold worked microstructure of the high-purity aluminum specimen.
The next example is the as-cast Al –7.12% Si alloy, prepared by

Surface	Abrasive Size	Load Lb (N)	Platen Speed/Direction	Time (min.)
CarbiMet	240-grit SiC water cooled	5 (22)	240 rpm Contra**	1 per sheet
UltraPol silk	9-µm MetaDi Diamond*	5 (22)	150 rpm Contra**	5
TriDent Polyester	3-µm MetaDi Diamond*	5 (22)	150 rpm Contra**	5
TriDent Polyester	1-µm MetaDi Diamond*	5 (22)	150 rpm Contra**	3
MicroCloth	0.05-µm MasterMet	5 (22)	150 rpm	3
MicroCloth	0.05-µm MasterMet	-	VibroMet2	≥20

* Add MetaDi Fluid lubricant (charge with paste and MetaDi Fluid, then add MetaDi Supreme suspension during the cycle)

** Contra means that the platen and the specimen holder rotate in opposite directions.

TAB. 1 ***Preparation Method for High-Purity Aluminum.***
Metodo di preparazione per alluminio di elevata purezza.

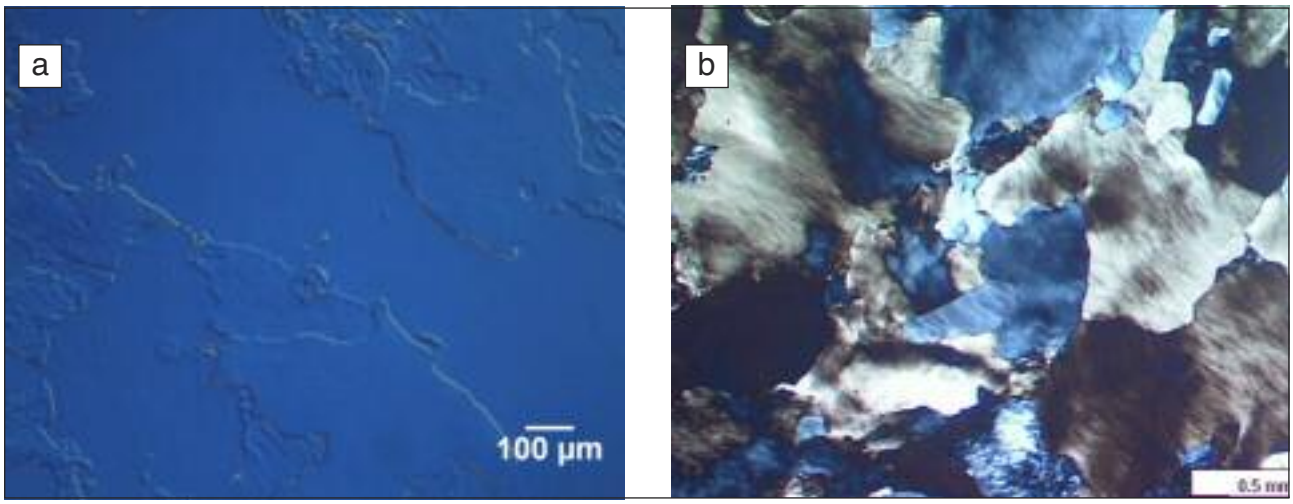

FIG. 1 ***Microstructure of cold worked 99.999% Al; a) Keller's reagent, Nomarski DIC; b) Barker's reagent, 20 V dc, 2 minutes, polarized light plus sensitive tint.***
Microstruttura di Al 99,999% lavorato a freddo; a) reagente di Keller Nomarski DIC; b) reagente di Barker, 20 V dc, 2 minuti, luce polarizzata più colorazione.

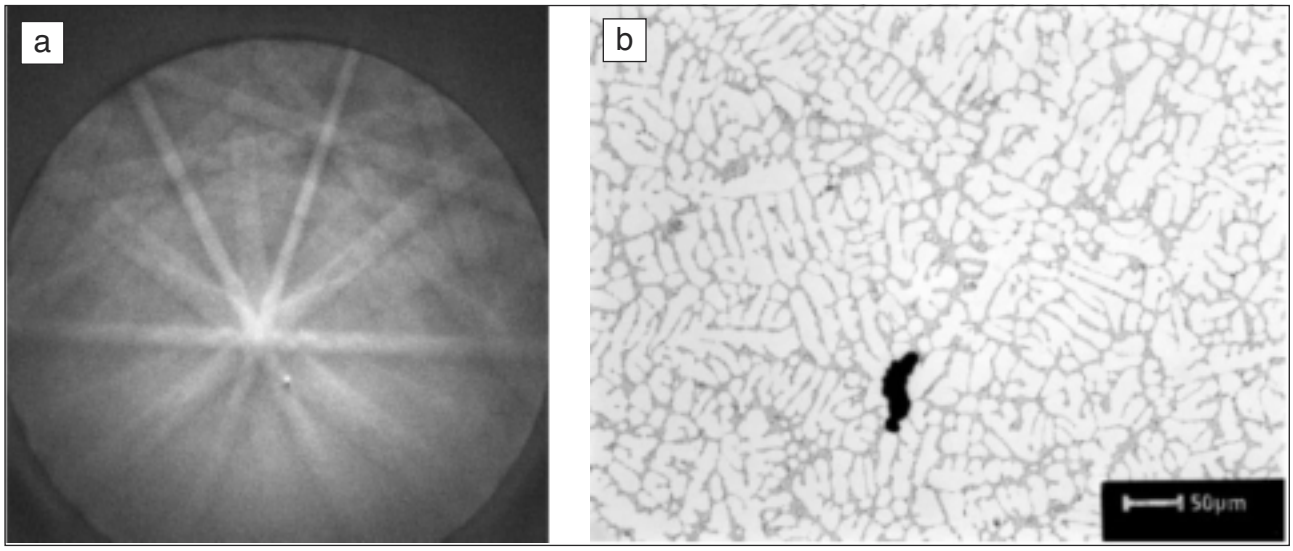

FIG. 2 ***a) EBSD pattern for α-Al in as-cast Al – 7.12% Si – pattern quality index: 87 ± 4.2;***
b) light micrograph of as-cast Al-7.12% hypoeutectic alloy etched with 0.5% HF in water.
*a) diagramma EBSD per **a**-Al in Al – 7.12% Si as-cast – indice di qualità del diagramma: 87 ± 4.2;*
b) micrografia di lega ipoeutettica Al-7.12% as-cast sottoposta ad attacco con 0.5% HF in acqua.

Surface	Abrasive/Size	Load lbs. (N)	Speed rpm/Direction	Time (min.)
CarbiMet	240 (P280) grit SiC water cooled	6 (27)	240 Contra	U.P.
UltraPol or TriDent cloths	9-µm MetaDi diamond*	6 (27)	150 Contra	5
TriDent or TexMet pads	3-µm MetaDi diamond*	6 (27)	150 Contra	5
TriDent or TexMet cloths	1-µm MetaDi diamond*	6 (27)	150 Contra	4
MicroCloth or ChemoMet pads	0.05-µm MasterMet Colloidal silica suspension	6 (27) (7 lb/31 N for) ChemoMet	150 Contra	3
MicroCloth	0.05-µm MasterMet	-	VibroMet2	≥20
* Add MetaDi Fluid lubricant (charge with paste and MetaDi Fluid, then add MetaDi Supreme suspension during the cycle)				

TAB. 2 ***Preparation Method for High-Purity Copper.***
Metodo di preparazione applicato al rame ad alta purezza.

the same five-step method, but with only 4 minutes for the 3-µm step, and without vibratory polishing. The as-cast microstructure consists of α-Al dendrites and a eutectic of α-Al and Si. The α-Al dendrites were sampled for the EBSD patterns. As can be seen in Figure 2, an excellent quality diffraction pattern was obtained from the alpha-Al dendrites. Figures 1 and 2 demonstrate that mechanical preparation is capable of producing high quality EBSD patterns when properly performed.

Pure copper is extremely ductile and malleable. Copper and its alloys come in a wide range of compositions, including several variants of nearly pure copper for electrical applications that are very difficult to prepare damage free. Rough sectioning and grinding practices can easily damage copper and its alloys and the depth of damage can be substantial. Scratch removal, particularly for pure copper and brass alloys, can be very difficult. If the scratches are not removed, there will be damage beneath. Following the preparation cycle with a brief vibratory polish using colloidal silica is very helpful for scratch and damage removal. Attack polishing additions have been used in the past to improve scratch removal but are not necessary using the contemporary method followed by vibratory polishing.

Table 2 lists a five-step method for preparing copper and its alloys (vibratory polishing is an optional 6th step). It is always helpful, particularly with alloys that are difficult to prepare damage free, to etch the specimen after the fifth step, and then repeat the fifth step. This reduces damage and gives better EBSD patterns. Figure 3 shows a combined EBSD grain orientation map plus index of quality map for tough-pitch copper (Cu with about 400

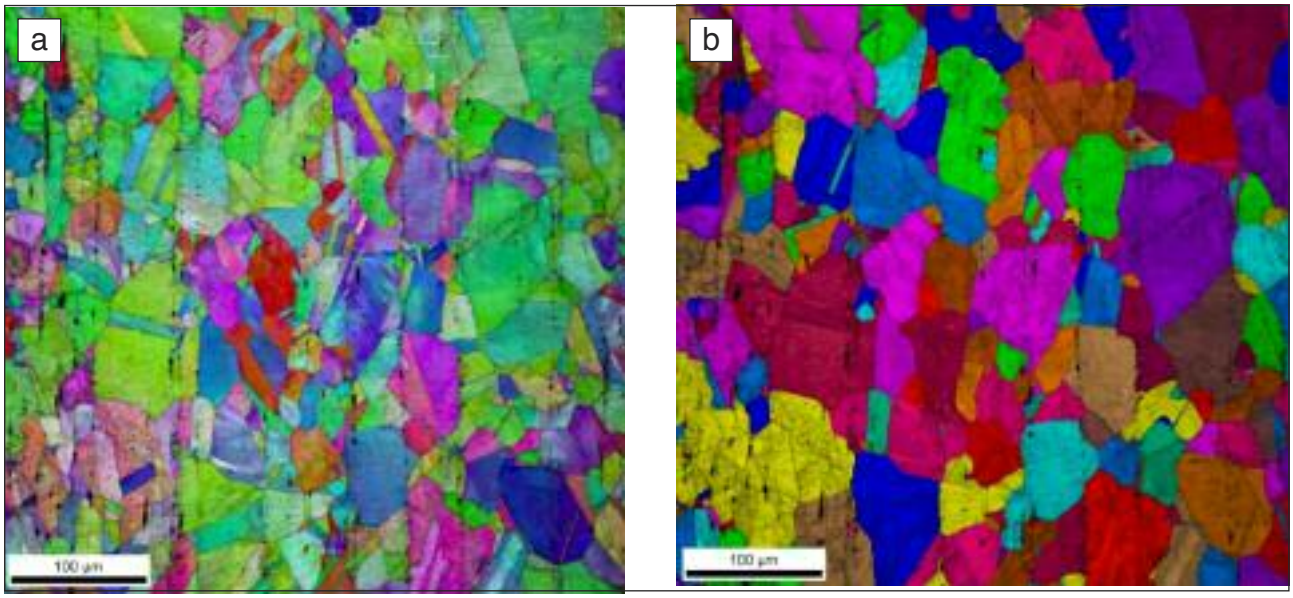

FIG. 3 ***EBSD grain orientation maps plus index of quality maps for tough-pitch copper; a) maps with twins; b) maps after twins were removed.***

Mappature dell'orientamento dei grani tramite EBSD e indice della qualità delle mappe per rame ETP; a) mappe con geminati; b) mappe dopo la rimozione dei geminati.

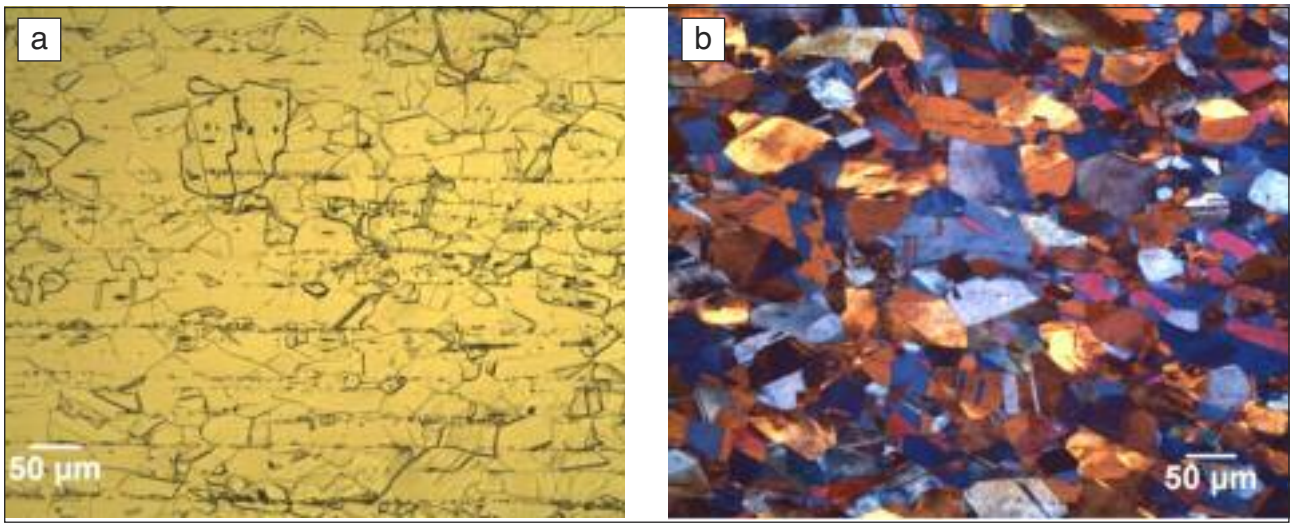

FIG. 4 ***Microstructure of wrought, annealed tough-pitch copper; a) etched with equal parts ammonium hydroxide and hydrogen peroxide (3% conc); b) Beraha's PbS tint etch, polarized light plus sensitive tint illumination.***

Microstruttura di rame ETP trafilato e ricotto; a) dopo attacco con parti uguali di idrossido di ammonio e perossido di idrogeno al 3%; b) con attacco colorante PbS di Beraha, luce polarizzata e illuminazione opportuna per la colorazione.

ppm oxygen) which reveals the grain structure and annealing twins. Figure 3 also shows the map after twins have been removed. Note that a few twins remained after image processing that will be removed if the boundary angle requirement for a twin is made slightly greater. This specimen was not etched. Figure 4 shows the specimen after etching for comparison. Measurement of grain size in twinned Cu and its alloys is nearly impossible by light microscopy image analysis due to the inability to reveal all of the grain boundaries and twin boundaries, except by color etching.

Figure 5 shows an EBSD pattern and the microstructure of wrought cartridge brass, Cu – 30% Zn, that was cold reduced 50% in thickness and then annealed at 704 °C for 30 minutes producing a coarse twinned α-Cu matrix. This is a relatively difficult alloy to prepare free of scratches and surface damage and the EBSD pattern quality was superb. The method shown in Table 2 was utilized to prepare this specimen except that the times for the 3- and 1-µm steps were 4 and 3 minutes, respectively, followed by a 30 minute vibratory polish.

EBSD patterns can be developed for both phases in a two-phase alloy, as long as preparation keeps both phases flat on the plane-of-polish. If relief is present, such that one phase is recessed below the surface, EBSD patterns will not be developed. As an example, a specimen of Naval Brass, an α-β brass consisting of Cu – 39.7% Zn – 0.8% Sn, was tested after etching which attacked the β phase. EBSD patterns could be generated from the

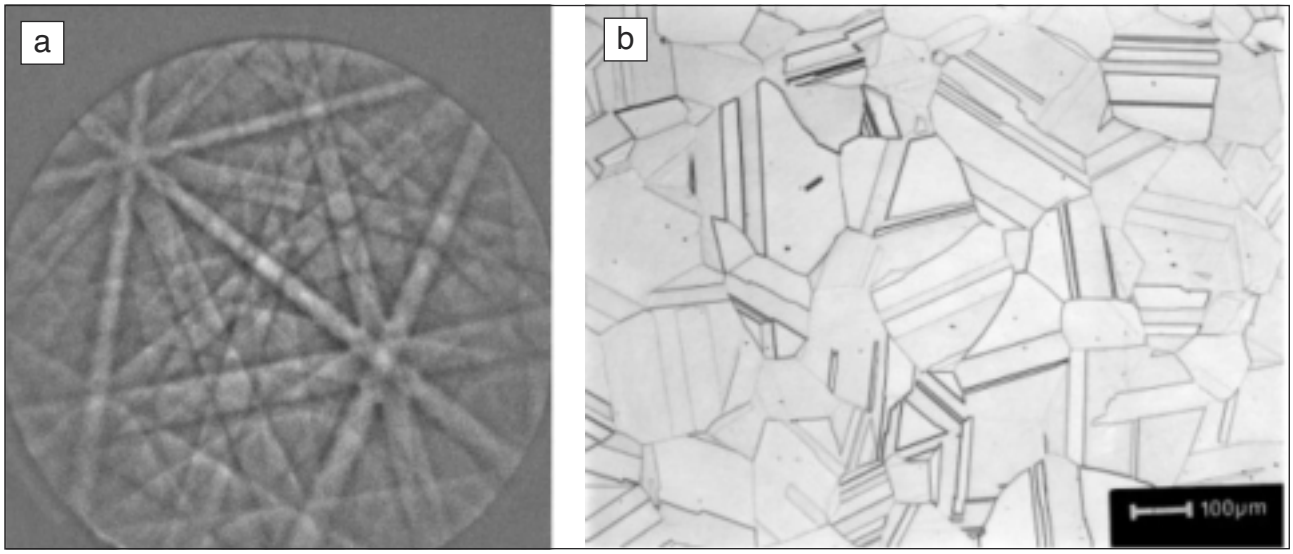

FIG. 5 ***EBSD pattern and microstructure of cartridge brass: a) EBSD pattern for Cu – 30% Zn – PQI: 221 ± 8.6; b) microstructure of wrought, annealed Cu – 30% Zn etched with equal parts hydrogen peroxide (3%) and ammonium hydroxide.***

Diagramma EBSD e microstruttura di ottone per munizioni; a) diagramma EBSD di Cu – 30% Zn – PQI: 221 ± 8.6; b) microstruttura di Cu – 30% Zn trafilato e ricotto sottoposto ad attacco con perossido di'idrogeno (3% conc) e idrossido di ammonio in parti uguali.

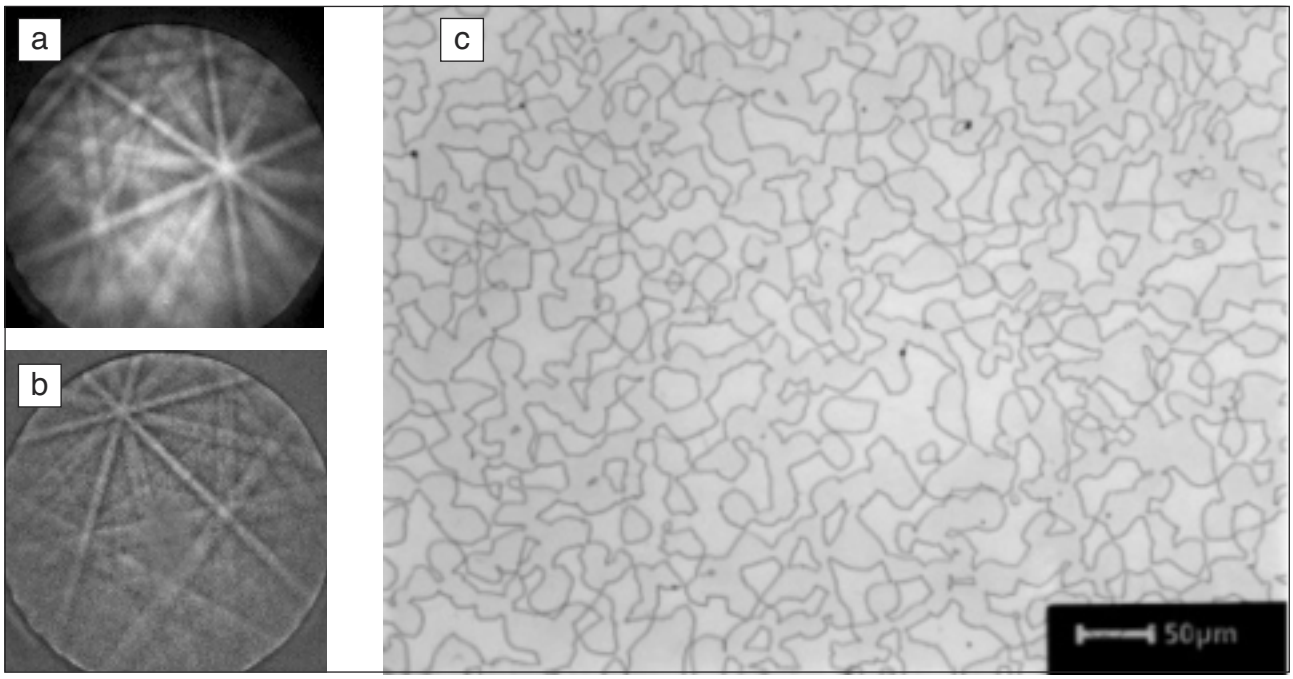

FIG. 6 ***EBSD patterns and microstructure of Naval Brass; a) and b): EBSD patterns for the alpha and beta phase with PQIs of 118.5 ± 8.7 for α-Cu and 150.4 ± 20.7 β-Cu; c) microstructure after etching with 100 mL water, 3 g ammonium persulfate, 1 mL ammonium hydroxide (α-Cu is the continuous phase).***

Diagramma EBSD e microstruttura di ottone navale; a) e b) diagramma EBSD delle fasi alfa e beta con indici PQI di 118.5 ± 8.7, per α-Cu e 150.4 ± 20.7, per β-Cu; c) microstruttura dopo attacco con 100 ml acqua, 3 g persolforato di ammonio, 1 ml idrossido di ammonio (fase continua α-Cu).

α phase, but not from the recessed β phase. Re-polishing and running the specimen unetched produced excellent results for both the α and β phases as shown in Figure 6. The specimen was prepared in the same manner as used for the cartridge brass specimen.

EBSD maps can be made using a number of techniques. Figure 7 shows a grain orientation map, an index of quality map, the combination of these two maps, and a grain-orientation map where the colors have been assigned based on crystal orientation using an inverse pole figure.

Perhaps the most difficult metals and alloys to prepare for EBSD have been zirconium and its alloys. Numerous approaches have

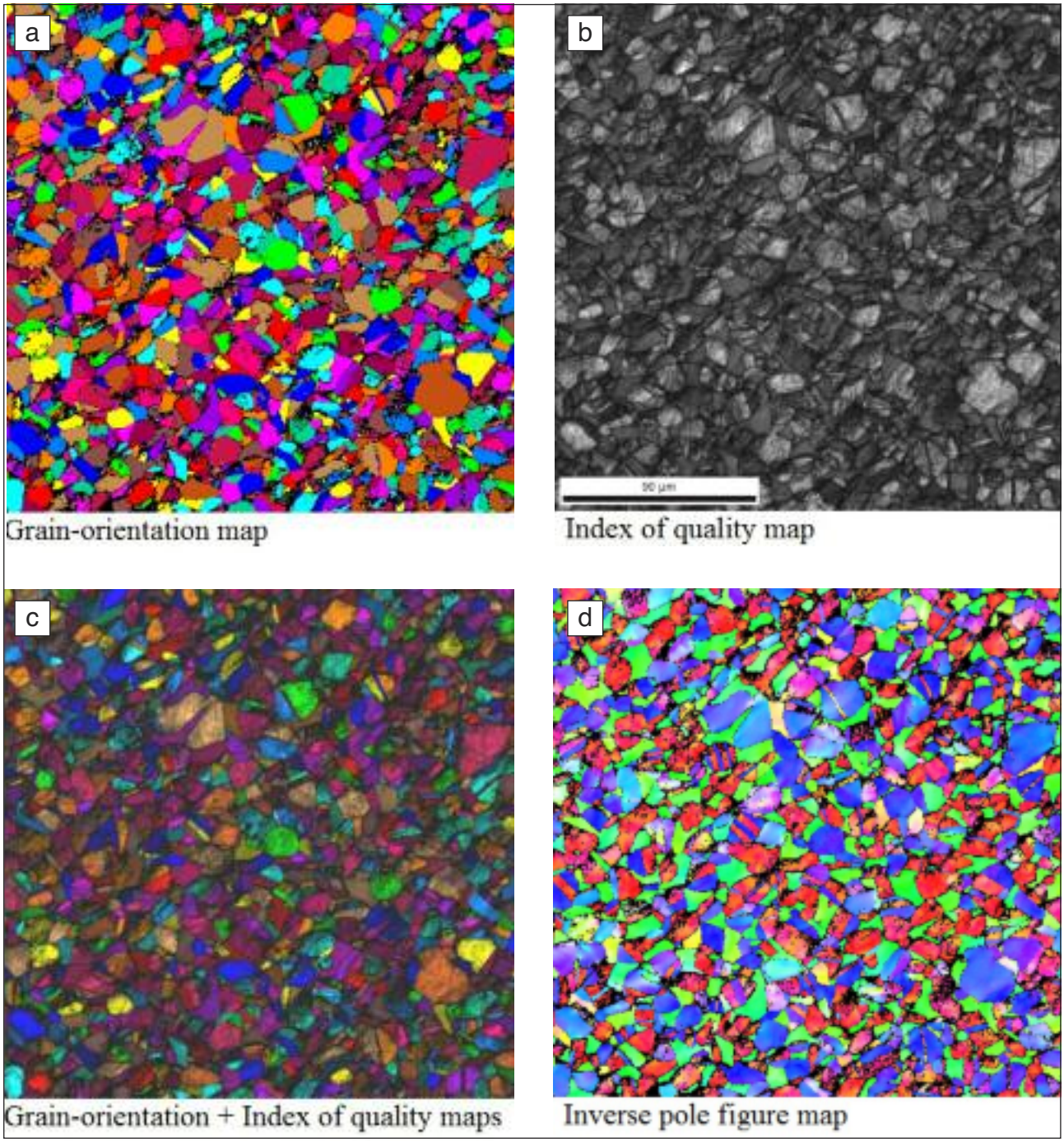

FIG. 7 ***Various EBSD maps for the Naval Brass specimen.***
Varie mappature EBSD per il provino di ottone navale.

been tried. Table 3 presents the method used that yielded excellent grain maps of high-purity Zr and Zr alloys. The SiC paper was coated with paraffin wax before grinding. Final polishing was performed using a 5 to 1 ratio of colloidal silica to hydrogen peroxide (30% conc.). In this experiment, the vibratory step was used (30 minutes).
Figure 8 shows two maps of high-purity (99.99%), annealed Zr. The first was constructed by adding an all Euler grain map with a band contrast map; the second shows an inverse pole figure map, plus grain boundaries, with the grains with missing pixels (black spots in the first map) filled in. The band contrast averaged 92.34 for the area shown.

Table 4 summarizes PQI results for a number of metals and alloys evaluated, many of which are difficult to prepare. These results clearly show that mechanical specimen preparation, if properly performed, is fully capable of producing damage-free surfaces that yield acceptable EBSD patterns that can be indexed reliably. The Ni-based superalloys (Carpenter's Custom Age 625 Plus and the fine-grained 718) contained sub-microscopic strengthening phases (the latter also contains copious delta phase) that make the EBSD analyses more difficult. The pure tantalum specimen was a P/M specimen that was not fully dense.
A second set of experiments evaluated the band contrast of eighteen (18) high-purity (generally >99.95%) specimens prepa-

Surface	Abrasive/Size	Load lbs. (N)	Speed rpm/Direction	Time (min.)
CarbiMet	240 (P280) grit SiC water cooled	5 (22)	240 Contra	U.P.
CarbiMet	320 (P400) grit SiC water cooled	5 (22)	240 Contra	1
UltraPol cloth	9-µm MetaDi diamond*	6 (27)	200 Contra	10
TriDent cloth	3-µm MetaDi diamond*	6 (27)	200 Contra	7
TriDent cloth	1-µm MetaDi diamond*	6 (27)	200 Contra	5
MicroCloth pad	0.05-µm MasterMet Colloidal silica suspension	6 (27)	200 Contra	7
MicroCloth	0.05- m MasterMet	-	VibroMet2	≥20

* Add MetaDi Fluid lubricant (charge with paste and MetaDi Fluid, then add MetaDi Supreme suspension during the cycle)

TAB. 3 ***Preparation Method for High-Purity Zr and Zr Alloys.***
Metodo di preparazione per Zr e leghe Zr ad alta purezza.

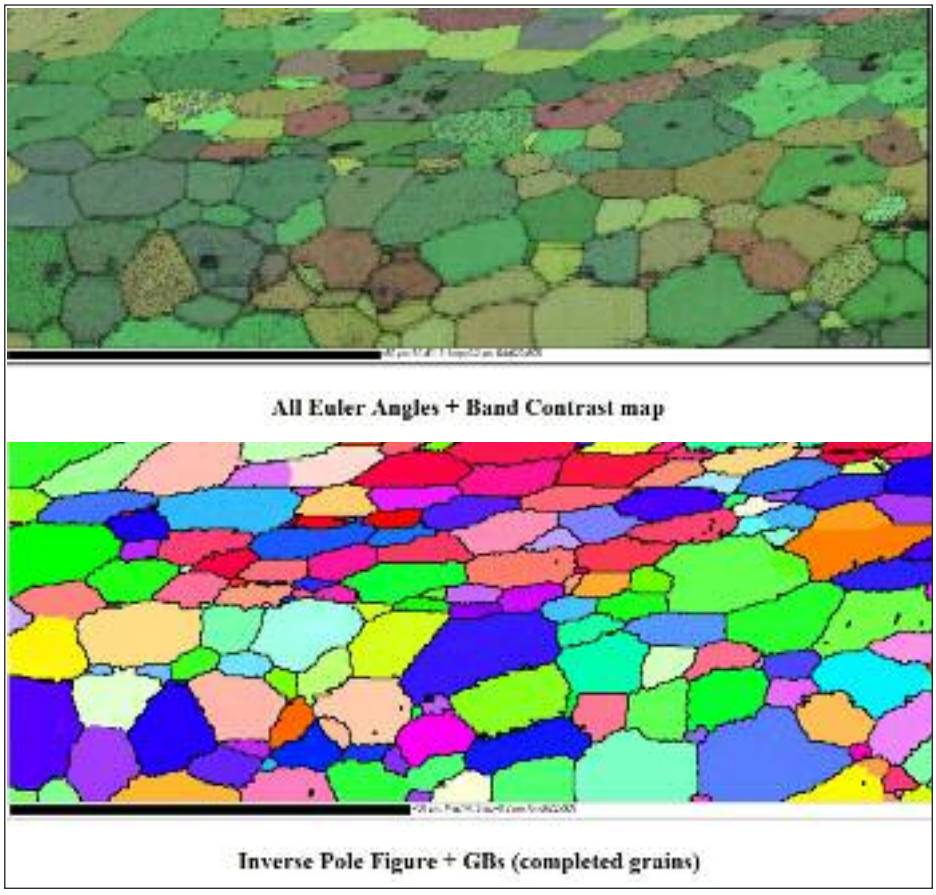

FIG. 8
Two examples of grain maps for high-purity (99.99%) Zr.
Due esempi di mappatura dei grani per Zr ad alta purezza (99.99%).

red using methods typical of those shown above, or similar methods, usually with five steps (four for Ti). These specimens varied from Mg (atomic number 12) to Bi (atomic number 83) and covered the range of metallic crystal structures: body-centered cubic (6), face-centered cubic (4), hexagonal close-packed (5), diamond cubic (1) and rhombohedral/trigonal (2). Table 6 lists the specimens prepared using our standard methods and analyzed. Results for six of these after vibratory polishing are shown in Table 5.

Specimens of pure Sb, V and Zr were susceptible to SiC embedment, even though the grit size was coarse, e.g., 240- and 320-grit. Hence, grinding was repeated after coating the paper with paraffin wax. Attack polishing was used, mainly with 30% conc. H_2O_2, for the last step for preparing Cr, Nb, Ti, W and Zr. MasterMet colloidal silica was used for the last step, except for preparing Fe (MasterPrep alumina was used) and Mg (water-free MasterPolish was used). Oil-based diamond suspensions (9-, 3- and 1-µm) were used to prepare the high-purity (99.999%) Mg. For the Bi and Pb pure specimens, grinding used four steps: 240-, 320-, 400- and 600-grit SiC paper coated with paraffin wax with low loads, followed by three polishing steps using 5-, 1- and 0.3-µm alumina slurries and a final polish with MasterMet colloidal silica. All polishing steps used MicroCloth synthetic suede cloth. Although the Bi produced an excellent EBSD pattern, none was obtained

Metal/Alloy	PQI ± 95% CL	Metal/Alloy	PQI ± 95% CL
α-Al in Al-7.12% Si	87 ± 4.2	α-Cu in Cu-30% Zn	221 ± 8.6
Cu–39.7% Zn–0.8% Sn	118.5 ± 8.7 for α	Cu–39.7% Zn–0.8% Sn	150.4 ± 20.7 for β
Elgiloy (Co-based)	221.4 ± 7.4	Pure Fe	249.6 ± 5.5
Si Core Fe B	199.9 ± 7.4	316 Stainless Steel	184.9 ± 8.5
2205 Duplex SS	248 ± 15.4 for α	2205 Duplex SS	207.9 ± 11 for γ
Ni-200	176.3 ± 17.6	HyMu 80 (Ni-base)	196.7 ± 7.2
Nitinol (Ni-Ti)	58.7 ± 4.3	CA625 Plus (Ni-base)	200.5 ± 6.5
Fine Grain 718 (Ni-base)	80.7 ± 4.4	Pure Cr	259.8 ± 13.1
Pure Nb	166.2 ± 17.1	Pure V	125.9 ± 10.3
Pure Ta	169.7 ± 13.0	CP Ti ASTM F67 Gr2	119.1 ± 4.1
W in W-27 Cu	296.9 ± 20.1	Pure Bi	86.2 ± 1.8
Pure Pb	49.3 ± 3.0	Pure Ru	266.2 ± 21.8

TAB. 4 ***Pattern Quality Index Values for Various Metals and Alloys.***
Valori dell'indice di qualità del diagramma per diversi metalli e leghe.

High-Purity Elements	Atomic Number	Crystal Structure	Band Contrast (0-255)
Mg	12	hcp	161.2
Al	13	fcc	151.2
Si	14	diamond cubic	205.75
Ti	22	hcp	134.0
V	23	bcc	102.2
Cr	24	bcc	88.27
Fe	26	bcc	105.4
Ni	28	fcc	85.0
Cu	29	fcc	122.6
Zn	30	hcp	170.8
Zr	40	hcp	77.3
Nb	41	bcc	145.6
Ru	44	hcp	66.0
Sb	51	rhombohedral	180.2
Ta	73	bcc	122.8
W	74	bcc	91.6
Pb	82	fcc	No Pattern
Bi	83	rhomb./trigonal	255

TAB. 5 ***Band Contrast Values for 18 Pure Metals.***
Valori della Banda di Contrasto per 18 metalli puri.

High-Purity Element	Mean Band Contrast (0 to 255)	
	Standard Method	Standard + Vibratory Polish
Mg	161.2	175.25 (+8.7%)
Si (single crystal)	205.75	233 (+13.2%)
Ti	134.0	146.2 (+9.1%)
Ni	85.0	102.8 (+20.9%)
Nb	145.6	151.2 (+3.8%)
Pb	No pattern	108.0

* A 60 minute vibratory polish was used for the lead specimen.

TAB. 6 ***Band Contrast Improvement Due to Vibratory Polishing (20 min.*).***
Miglioramento della Banda di Contrasto dovuta a lucidatura a vibrazioni (20 min.).*

with the pure Pb specimen. A one-hour vibratory polish with MasterMet colloidal silica using a MicroCloth pad was required to obtain a diffraction pattern for Pb.
A two-minute chemical polish is normally used after mechanical polishing of Zr; so EBSD was conducted on a second specimen after chemical polishing. Surprisingly, no pattern could be obtained on the chemically polished specimen. The chemical polish improved polarized light response but introduced grain faceting (excessive relief). It has been reported that using heavy pressure with the same chemical polish minimized relief and yielded good EBSD grain maps. The result for pure Zr in Table 5 was obtained on the same specimen as illustrated above in Figure 7, but after an earlier preparation attempt with a less effective preparation method than presented in Table 3. The average band contrast for the high-purity Zr specimen using the method in Table 3 was 92.34 and ~90% of the pixels produced indexable diffraction patterns. For the results published in Table 5, the average band contrast was 77.3 and only about 20% of the pixels yielded indexable diffraction patterns.
Five specimens were evaluated after our standard preparation method and then after a subsequent 20 minute vibratory polish to determine the degree of improvement that can be obtained. If the method used to prepare the specimens is not as good as what was used in our work, then the vibratory polish will produce a greater improvement. Longer times will also yield greater improvements. Table 6 summarizes these test results. Vibratory polishing improved the band contrast of the first five elements tried by an average of 11.1%; patterns could not even be obtained with lead without a vibratory polish.
Details on the preparation methods used to prepare these alloys, and others, can be obtained from the author or at the web site: www.buehler.com.

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Abstract

Preparazione di provini metallografici per diffrazione con elettroni retrodiffusi

Parole chiave:
metallografia, microscopia elettronica, prove

La Diffrazione con elettroni retrodiffusi (EBSD) viene realizzata con il microscopio elettronico a scansione (SEM) per fornire un’ampia gamma di dati analitici connessi, ad esempio, agli studi dell’ orientamento cristallografico, all’identificazione delle fasi e delle dimensioni dei grani. Un diagramma di diffrazione può essere ottenuto in meno di un secondo, ma si può migliorare la qualità dell'immagine utilizzando un tempo di scansione più lungo. La mappatura dei grani richiede lo sviluppo di diagrammi di diffrazione per ogni pixel nel campo e si tratta di un processo più lento. La qualità del diagramma di diffrazione, che influenza la affidabilità dell’indicazione contenuta nel diagramma stesso, dipende dall’eliminazione del danneggiamento del reticolo dovuta alla preparazione dei provini. E 'stato affermato che la rimozione di tale danneggiamento può essere ottenuta solo con la lucidatura elettrolitica o la lucidatura a fascio ionico. Tuttavia, l'uso dei moderni metodi di preparazione meccanica, delle attrezzature e dei materiali attualmente disponibili rende possibile la produzione di immagini di diffrazione con eccellente qualità senza dover ricorrere all’uso di elettroliti pericolosi e senza i problemi e i limiti connessi con la lucidatura elettrolitica e con fascio ionico.
In pratica si otterranno diffrazioni EBSD con indici di alta qualità se, successivamente alle preparazioni meccaniche, risulta possibile ottenere immagini di qualità con la luce polarizzata - nel caso di elementi e leghe a struttura cristallina non cubica (ad esempio, Sb, Be, Hf, α-Ti, Zn, Zr) - oppure risulta possibile produrre immagini a colori di alta qualità a seguito di attacchi chimici coloranti - nel caso di elementi o leghe a struttura cristallina sia cubica che non cubica - ; si ottiene così una verifica del fatto che la superficie è priva degli effetti nocivi di un danneggiamento da preparazione. Per ottenere i risultati migliori è necessario inoltre avere un’ eccezionale planarità della superficie, a causa dell’angolo acuto tra il provino e il fascio di elettroni (70 - 74°).
I procedimenti di preparazione dei provini sono dunque fondamentali e sono state messe a punto sequenze di operazioni - sia per i metalli che per le leghe - semplici e di breve durata (dell’ordine dei 25 minuti). La prima applicazione qui presentata riguarda l’alluminio ad alta purezza (99.999%), deformato e incrudito. L’alluminio in queste condizioni presenta difficoltà particolari per l’esecuzione della diffrazioni EBSD a causa del basso numero atomico che implica difficoltà nel generare elettroni retrodiffusi; inoltre va considerato che i metalli ad alta purezza sono di per sé difficili da preparare, soprattutto se incruditi e non ricristallizzati, quindi con distorsioni del reticolo cristallino. La procedura messa a punto per l’alluminio è stata poi applicata alla lega per fonderia Al – 7.12 % Si, bifasica e allo stato “come fuso”.
Tenuto conto che il taglio e la preparazione metallografica con abrasivi può danneggiare in profondità materiali metallici duttili e malleabili, sono state poi indagati il rame e le sue leghe, in particolare rame ETP (Electrolitic Tough Pitch), un ottone Cu – 30% Zn (deformato al 50% e ricotto), un ottone navale Cu – 39.7% Zn – 0.8% Sn, bifasico. Per quest’ultimo si è proceduto ad una doppia indagine: solo sulla fase α (dopo attacco selettivo della fase β) e successivamente su entrambe le fasi. Una seconda sequenza di indagine ha riguardato 18 provini costituiti da elementi ad alta purezza (generalmente >99,95%), che andavano, come numero atomico, dal magnesio al bismuto e come reticolo cristallino dal cubico a corpo o a facce centrate all’esagonale compatto e al romboedrico/trigonale.