TECHNotes

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How to Select and Use Final Polishing Oxide Media

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Suspension Selection Guide

Final polishing suspensions are designed to remove the final layer of surface deformation that is left after diamond polishing steps. While this deformation is often invisible to the naked eye, it must be removed when conducting high end microscopical analysis. A variety of oxide polishing media are available to suit different material types and the analysis desired after polishing. The table below summarises the selection criteria for different oxide polishing media that Buehler supplies.

MasterPrep Alumina	MasterMet Colloidal Silica	MasterMet 2 Colloidal Silica	MasterPolish Final Polish
			
Sol-gel alumina suspension	Amorphous colloidal silica suspension	Non-crystallizing amorphous colloidal silica suspension	Blend of high purity alumina and colloidal silica suspension
0.05µm	0.06µm	0.02µm	0.05µm
~8.5pH	~10pH	~10.5pH	~9pH
Mechanical polishing action	Chemo-mechanical polishing action	Chemo-mechanical polishing action, suitable for automatic dispensing	Chemo-mechanical polishing action. With minimal water content for water sensitive material
Excellent for minerals, ferrous metals, low melting point alloys, carbides, PWB's, precious metals, electronics, polymers, and polymer matrix composites	Excellent for metals, minerals, ceramics, and polymers	Excellent for metals, minerals, ceramics, and polymers	Excellent for most magnesium alloys, cobalt alloys, most iron alloys, nickel, and metal matrix composites

MasterPolish 2 Final Polish	MicroPolish Alumina Suspension	MicroPolish II Alumina Powder
		
Iron oxide suspension	Agglomerated alumina	High quality deagglomerated alumina
0.06µm	1µm, 0.3µm, 0.05µm	0.3µm
~10pH	~8.5pH	~8.5pH
Chemo-mechanical polishing action	Mechanical polishing action	Mechanical polishing action
Excellent for sapphire, glass, alumina, silicon nitride, and metal/ ceramic composites	Offering high removal rates and good for use on magnesium, lead, and their alloys	Offering better surface finish and suitable for most minerals & metals.

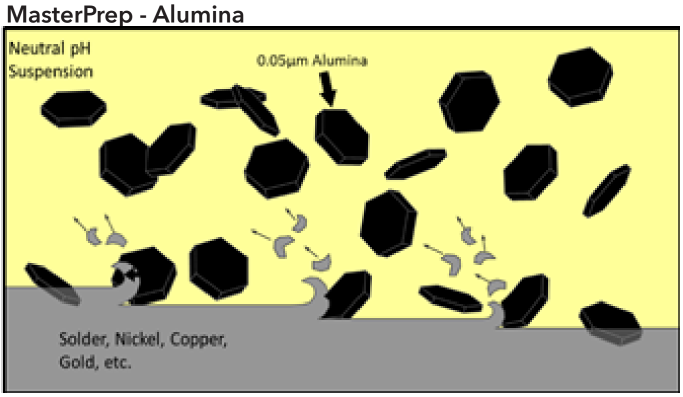
Alumina or Silica?

Alumina is commonly used as an abrasive particle for final polishing of materials. Its method of production has an influence on the type of alumina, and how it's used during polishing steps. This can be via a fusion production process, and then crushed to appropriate grit and graded. Alumina can also be produced via a calcining process where better control of the particle size is achieved offering finer abrasive alumina, but calcining process results in mixtures of alpha alumina and gamma alumina. These mixtures in use tend to agglomerate but also do break up during use. For alumina suspension that does not agglomerate, a sol gel process is used to manufacture the suspension with the added advantage of the ability to produce smaller particles sizes, of up to 1/10th of equivalent crushed alumina.

Silica on the other hand comes in amorphous form as a colloidal dispersion. The dispersion contains submicron level silica particles in a glycerine-based solution, that also has a carboxyl polymethylamine polymer added. The resultant mixture normally has a pH of around 10, with the silica particles exhibiting a spherical shape of approximately 0.01 to 0.09 microns as opposed to multifaceted surface structures observed on alumina particles. The addition of polymer in silica suspension stabilizes the dispersion delaying evaporation and lowering its freezing temperature. Without the polymer addition, the silica will crystallise with subsequent loss of it polishing performance.

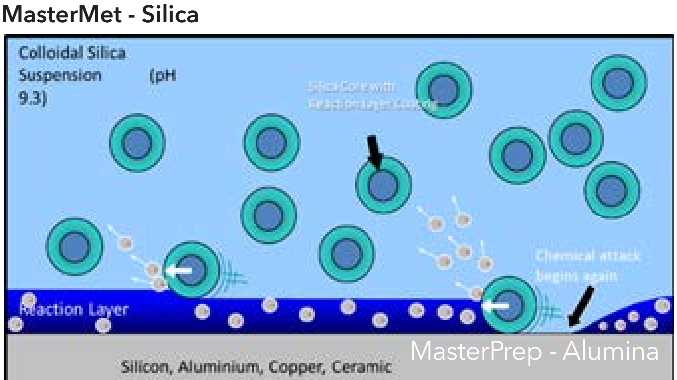
What is the mechanism of material removal?

MasterPrep - Alumina



Alumina removes material through mechanical, abrasive process
Due to a multifaceted particle structure like diamond abrasive particles, alumina particle remove material through a shear mechanical process as illustrated.

MasterMet - Silica



Silica removes materials through a chemo-mechanical process
Silica on the other hand appears as spherical particles unlike diamond or alumina, and the material removal process has a two-fold approach, a rolling action to 'tickle" the surface and due to its high pH, the suspension does surface dissolution of the surface being polished.

MasterPolish
The suspension offers mechanical material removal properties of alumina and the chemo mechanical properties of silica in a carefully controlled mix for water sensitive materials.

Tip
For mixed materials, and certain ferrous and non-ferrous materials, a 50% MasterPrep with 50% MasterMet mix will offer a more effective polishing suspension that benefits from both mechanical and chemo-mechanical effect of alumina and silica.

What is the surface finish like?

The micrographs illustrate the surface finish after polishing an electronic component solder region. Figure 1(a) shows resultant alumina polish surface without scratches and the copper interconnects on either side of the solder, whereas, Figure 1(b) shows silica polish finish with the solder region exhibiting more microstructural details. If one were to investigate defects such as porosity, voids within the solder region, MasterPrep (alumina) would be the best choice as shown in Figure 1(a).. If the analysis requires microstructural investigation looking at interdiffusion region between the solder and copper interconnects or the dendritic structure from cooling of the solder, then MasterMet (silica) would be preferred suspension to use , as illustrated in Figure 1(b).

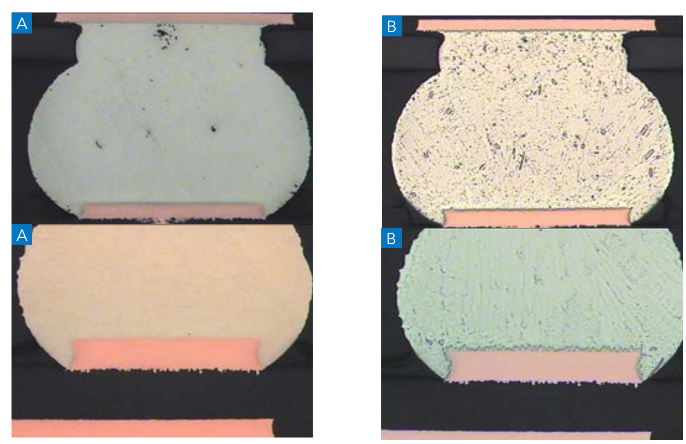


***Figure 1**. Electronic Component - Solder; (a) Ideal for geometrical measurements, voids/porosity inspections (b) Ideal for microstructural analysis, Interfacial analysis*

How about particle size on surface finish?

Abrasive particle size of suspension affects how long it takes to remove all the remnant scratches or deformation on a sample. Figure 3 (a) and (b) illustrate polishing action of using 2 different abrasive particle size on colloidal silica suspension MasterMet. One can easily discern presence of scratches on the surface in figure (b), polished with 20nm silica particles compared to figure (a) where 60nm particles size have been used to polish the surface. The samples were polished for 1 minute 30 seconds each using the same polishing parameters (force, speeds and cloth surface). The results show that using larger particle size does speed up the material removal process.

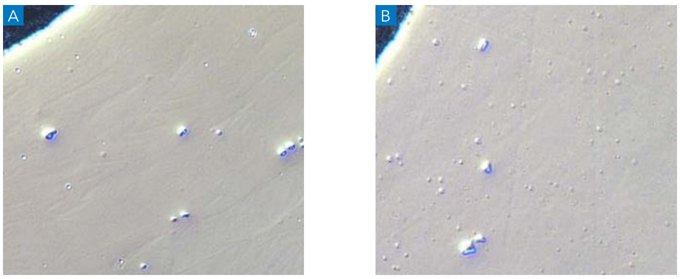


Figure 2. *(a) Silica MasterMet 60nm particle size, (b) Silica MasterMet 2 20nm particle size*

In practice, we tend to use both for different surface finish requirements. If one were to conduct high magnification microscopical analysis such as electron back scatter diffraction analysis, the initial polishing regime may incorporate running the samples using MasterMet for 2 to 3 minutes as a final step, and then carrying out a longer polish session using MasterMet 2 on a vibratory polisher to remove any remaining superficial deformed layers on samples.

Additional considerations – Does it crystallise?

Final polishing suspensions also present a challenge relating to crystallisation effect during use. As previously highlighted, these suspensions tend to evaporate due to lower freezing temperatures and can form crystals on a polished surface that are difficult to remove. This is common with general purpose colloidal silica suspension such as MasterMet, however, there are non-crystallising variants such as MasterMet 2. As a guide during use, amorphous colloidal suspension demands that samples are cleaned immediately after polishing actio with plenty of water to prevent recrystallisation. Non-crystallising colloidal silica offers a wider operating window for cleaning operating window without worry of silica crystallisation and does present certain advantages during use.

MasterMet - crystallises	MasterMet 2 – non crystallising
- Clean immediately - Highly recommended to preclean during last 10-20 sec of polishing cycle. - Ideal for short polishing times, less than 3 minutes	- Easier to handle during polishing - Ideal for automatic suspension dispensing system (Burst system) - Ideal for long polishing steps applications like Titanium (6-10 min) - Vibropolishing methods (20-240 min)

Figure 3(a) shows recrystallisation effect of colloidal silica on as-polished surface a couple of seconds after polishing with slow cleaning. The suspension has formed a glassy layer on the surface that tends to crack up during subsequent cleaning causing minor scratches as well as introducing Silica contaminant on the surface.

Figure 3(b) shows suspension droplets left for a couple of minutes to further illustrate the evaporation effect and subsequent recrystallisation. Both MasterMet and MasterPrep will crystallise and its recommended to clean immediately after polishing and observe the 10-15 seconds flushing of the surface with water. Flushing the surface with water also aids the cloth surface cleaning ensuring a better cloth lifetime.

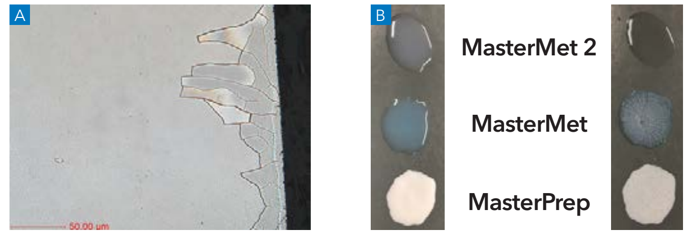


Figure 3. *(a) Recrystallised silica on sample surface with slow cleaning, (b) Micrographs illustrating suspension after dispensing, and when left to dry after 1 to 2 minutes*

What about Alumina powder Selection?

Alumina powders are selected when existing oxide suspension base is not suitable for the samples being prepared. MicroPolish I and II are made out of hard alpha alumina that gives efficient material removal combined with a superior surface finish. The powders are further classified as agglomerated or deagglomerated powder. Agglomerated powders tend to form lumps(agglomerates), due to electrostatic attraction between alumina powders, which can be broken down during the polishing process and generally have higher material removal rates compared to deagglomerated powder. However, deagglomerated powder is ideal for soft/ductile materials as it does not form lumps during use that would otherwise cause deeper subsurface deformation for an equivalent grit of agglomerated version.

Due to the inert nature of alumina, the powders are ideal for creating suspensions using non-aqueous carrier fluids making them ideal for polishing applications where the presence of water causes a reaction with the samples. MasterPolish suspension is designed for water sensitive samples and can be used in many cases. Certain samples require a close control of the oxide suspensions pH to minimise electrochemical activity of the phases present in these materials. An example is galvanised steel that can show selective etching during polishing. To minimise this, a suspension can be made with a pH as close as possible to 7 or neutral by adding mild alkalis or use of deionised water to create the slurry.

For water free suspensions, alumina powders can be mixed with non-aqueous carrier media such as propylene glycol or light hydrocarbon based polishing oils. Also note that use of oils might dramatically reduce the polishing rate.

A key consideration for alumina powders/suspension during use include

- The pH, a higher pH will decrease the polishing rate, ensure this is kept as inert as possible

- Use of soapy reagents, such as detergents will reduce alumina polishing effectiveness. Soapy detergents cause a fine frothy surface to develop affecting lubricity. on the other hand, addition of viscous soap solutions show a beneficial effect.

How much should you use?

Oxide polishing suspensions are generally used on a soft cloth with or without nap such as Microfloc, MicroCloth, TexMet-C or ChemoMet as examples. The ideal usage condition is to have the cloth surface sufficiently wetted out with the suspension to prevent likelihood of drying out. Soft and highly napped cloths are therefore desirable as these retain enough fluid during use to ensure the polishing action is optimal. Figure 4(a) and (b) illustrate ideal dosage conditions (wettability) on the cloth to ensure optimal polishing performance. Oxide Suspensions must be used in significant quantity as illustrated:

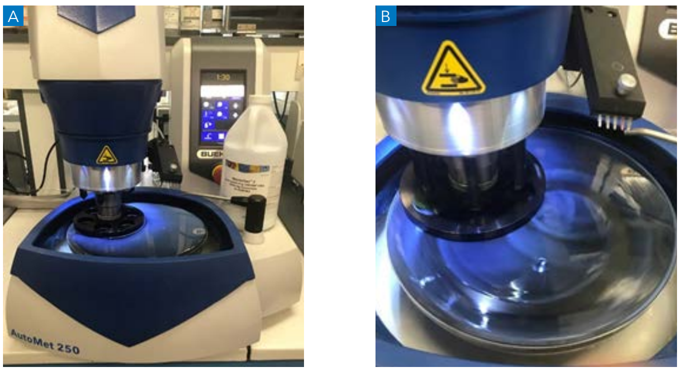


***Figure 5**. ChemoMet (soft porous) cloth dispensed with MasterMet 2 – ideal dispensing conditions*

Cleaning and storage considerations?

It is highly recommended to pre-clean samples and polishing cloth during the last 10-15 seconds of the final polishing step by flushing the surface with water as illustrated in Figure 5 (a) to (c). Flushing the surface with water aids to remove oxide polishing media from the cloth surface and samples. This has the advantage of ensuring repeatability in polishing as the cloth surface is clean but also improves the cloth lifetime.

Before storing the polishing cloth, an additional manual cleaning with water is carried out. Final manual cleaning can be done with the aid of a small plastic scrapper or a wooden stirring stick to wipe off remnant oxide suspension as the water rinses off on the rotating cloth (preferably at less than or equal to 100rpm). The cloth can be be finally dried on a stand before storage.

***Figure 4**. (a) just before last 20seconds (b) flushing surface with water, and (c) last 2-3 seconds*

When using alumina suspension and powder, the cloth surface will still exhibit presence of alumina particles entrapped in the cloth weave or porous structure. This is expected even with good cleaning, figure 6(d), after the final polishing stage. It is therefore good practice to reclean the cloth with water and carry out a rinse spin process before use especially if the cloth was dry before the final step.

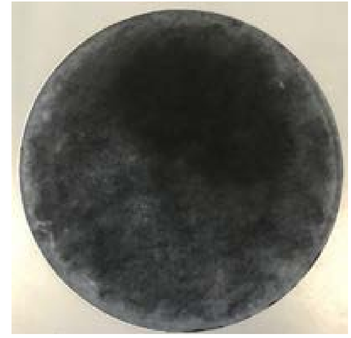


***Figure 6**. dry cloth after cleaning*

Therefore, it's better to also reclean the cloth with water and spin it before each use.

Storage

Suspensions:

- The suspensions should be stored in a cool and dry place. Direct sunlight should be avoided.
- Alumina suspensions, such as MasterPrep will undergo sedimentation when not in use. It is recommended to shake well before use.
- It is also recommended to clean the bottle opening before using the suspension. Due to recrystallisation effect of oxide polishing media, the bottle opening and/or bottle cap will exhibit crystallised particles that can cause scratches during use.

Polishing Cloths:

- Polishing cloths must be carefully stored to prevent debris or particles from contaminating the polishing surface.
- Avoid leaving the cloth on work benches, store away while not in use
- Have an allocated storage cabinet for your cloths.

Guidance on how long you should polish

For most metallic materials, a 2-minute polishing step is sufficient and optimal. There is a good balance on minimising polishing artefacts such as edge rounding even with low force application. Longer duration can be ideal for certain materials, however, edge rounding on microstructural feature become pronounced. For ceramic materials, longer polishing times are recommended as the materials are considerable harder compared to metallic materials.

Also note that larger scratches brought forward from preceding steps will not be removed in this step.

How about vibratory polishing VibroMet™ 2

Vibratory polishing is normally carried out to remove remaining surface deformation following a mechanical polish on a grinder polisher. This can be carried out on most materials, and the process involves running samples on a vibratory polisher, for at least half an hour using alumina or silica suspension. it is worth noting that the polishing rates on a vibratory polisher is slower compared to traditional grinder polishers and will require long polishing times depending on the material type. The most commonly used suspension is silica (preferably noncrystallising type, MasterMet 2) on a napped cloth.

How about attack polish of samples?

For certain materials, oxide polishing suspension can be mixed with etchants to aid with surface dissolution of difficult to remove subsurface deformed regions. This is applicable to hexagonal close packed (HCP) materials such as Titanium, Zirconium, hafnium among others and the procedure involves the addition of etching reagents onto silica-based oxide suspensions(mainly) or alumina-based suspension, referred to as attack polishing process.

Some common applications – refer to SumMet guide

- Titanium alloys
  - The addition of 1 part 30% Hydrogen Peroxide to 5 parts Silica (MasterMet or MasterMet 2),
  - or 1-part Ammonium Persulfate solutions (10g Ammonium Persulfate per 100mℓ distilled water) to 5 parts Silica (MasterMet or MasterMet 2).
- Zirconium /Hafnium alloys
  - The addition of 1-2 parts 30% Hydrogen Peroxide to 8 parts Silica (MasterMet or MasterMet 2)
  - 5ml chromium trioxide added to 95ml of MasterMet or MasterPrep.
  - Additions of oxalic, nitric acids have also been used.
- Copper alloys
  - Add a solution of ammonium hydroxide or copper ammonium persulfate solution.
  - The addition of 1 part 30% Hydrogen Peroxide to 5 parts Silica (MasterMet or MasterMet 2).
- Lead alloys
  - Add ammonium acetate (1-5gm) per 1000ml of MasterPrep.
- Refractory metals
  - A good general-purpose attach polish consists of 5ml chromium trioxide added to 95ml of MasterMet.

Useful References

SumMet, B. (2018). The Science Behind Materials preparation. Waukegan, Illinois, U.S.A. Retrieved from https://www.buehler.com/literature.php

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