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Preparing and Etching Titanium to Document Surface Affects

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Because of its properties, titanium is one of the more difficult materials to prepare for metallographic examination. [1] [2] This presentation is intended to further illustrate this point. The challenge is to understand and minimize preparation artifacts. Artifacts can be introduced if the wrong preparation steps are taken. For example if one used the same metallographic process used on a body centered cubic martensitic steel, then the etched microstructure would not be a true representation of titanium. This presentation will cover: 1) etchant selection and exposure times, 2) effects of mechanical deformation and cold work, 3) heat affected zones and 4) alpha case, a surface enrichment due to oxygen contamination during thermal exposure at temperatures >~540°C. Micrographs and some of the terminology and processes discussed in this presentation can be found in published literature. [1] [2]

All materials have unique properties which should be taken into account when preparing and etching cross-sections to bring out its microstructure. While similar to some materials, titanium is one of the most difficult to bring out its microstructure. It is very difficult to not introduce cold work and heat distortion during the sample preparation. There are also a variety of challenges with using etchants with titanium including application and exposure time of the etchant. For an example, one can etch the base microstructure and hide the hydrides A specialized etch is required for hydrides.

There is no tolerance for not having a properly prepared sample free of preparation-induced artifacts such as a deformation layer or one caused by heating during preparation which will mask the true microstructure. Selection of the etchant, etchant times, and etchant application method will all impact the final results. Most etchants should be applied by immersing the reagent on to the sample. A chem-milling etchant like Kroll's will work either by swabbing or an immersion (dip). A tint etchant is one that depends on the etching products to remain on the sample, which will align with the grain direction. After application, the tint etchant is very fragile and easily disturbed. If the etchant is swabbed with a brush for example, it will alter and possibly destroy the appearance. After applying the tint etch, just rinse the specimen off with tap water. Do not brush, swab, or wipe off the surface, just rinse and blow dry. With a tint etchant and polarized light the grain aliment will add to or depolarize the light, giving a highly contrasting microstructure.

If the specimen doesn't etch it is likely due to sample preparation. The chem-milling etchant feature has an optimum removal depth not to exceed the depth of field of the objective lens, 400 to 1200 microns. Tint etchant products also have an optimum depth build up. If the etch is applied too long, grain contrast will start to fade. The micrographs in Figure 1 demonstrate the clarity differences of the alpha case due to oxygen contamination observed on Ti surfaces, etched with a tint or chem milling etch (Krolls).

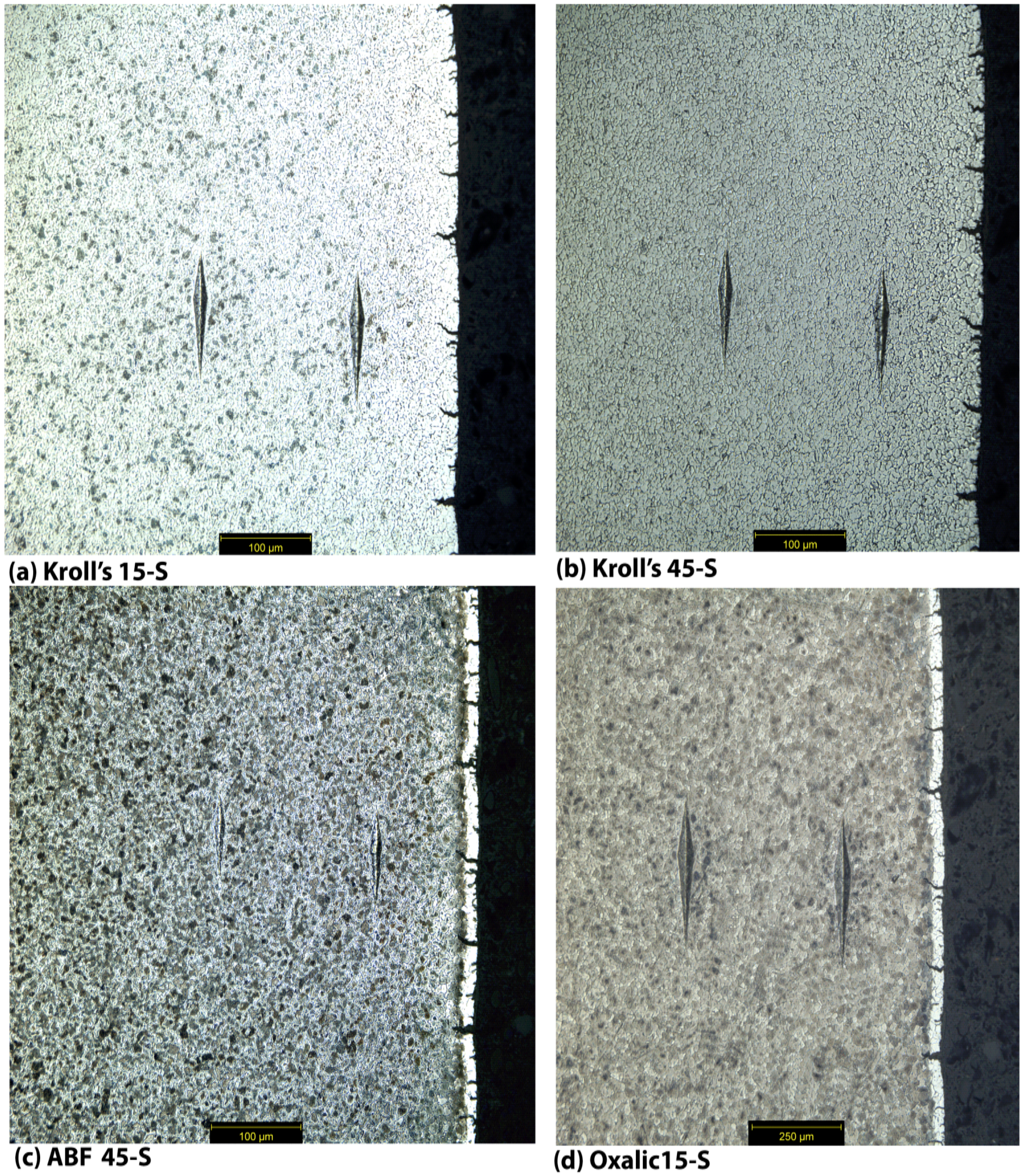

Figure 1 a,b,c & d: Ti-6Al-4V etched as indicated, with the number-s being the etch time in seconds. These micrographs demonstrate that the ABF and Oxalic etches/tints will normally show an alpha case much more clearly than the Kroll's etch. Kroll's will normally show it, but the difference between the alpha-case and matrix is often much more subtle as shown in the figure. Cracks are seen for more extreme cases.

REFERENCES

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