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Chemical Etching

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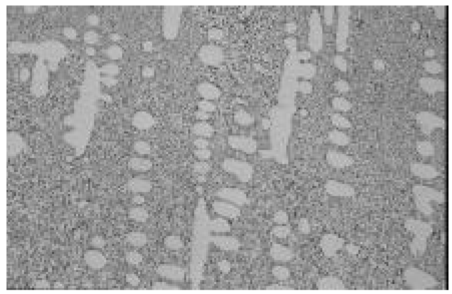
Etching - Enhancing Microstructures

The purpose of etching is to optically enhance microstructural features such as grain size and phase features. Etching selectively alters these microstructural features based on composition, stress, or crystal structure. The most common technique for etching is selective chemical etching and numerous formulations have been used over the years. Other techniques such as molten salt, electrolytic, thermal and plasma etching have also found specialized applications.



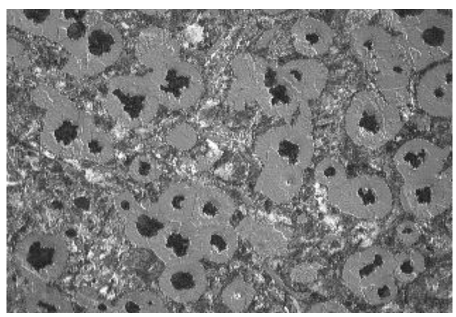
Aluminum-Silicon Alloy, B.F. 200X, Kellers etchant

Etching Basics

Chemical etching slectively attacks specific microstructural features. It generally consists of a mixture of acids or bases with oxidizing or reducing agents. To understand the basics of chemical etching, the relationship between pH and Eh (oxidation/reduction potentials), often known as Eh-pH diagrams or Pourbaix diagrams can be used.

Etchant Procedures

Specimen Preparation - proper metallographic specimen preparation is required to avoid artifacts such as an altered grain structure or a smeared microstructural surface.



Cast Iron, B.F. 100X, 2% Nital

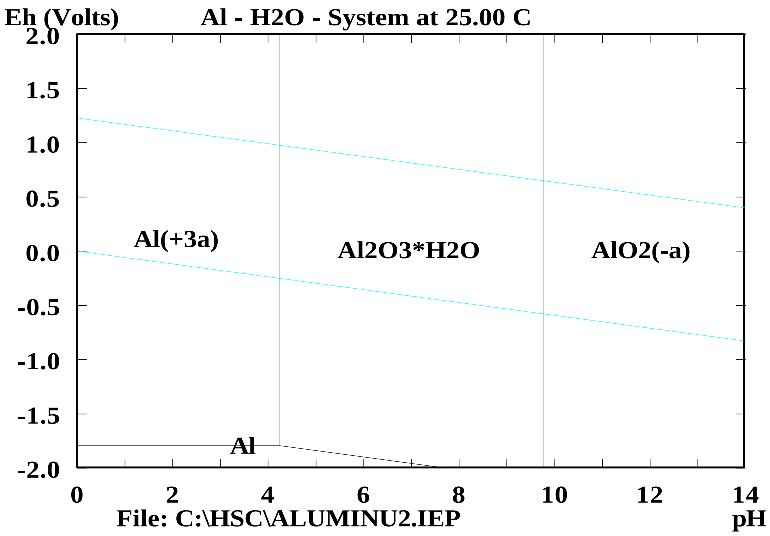
Common etching techniques include swabing and immersion. Swabing is less aggressive for more controlled attack, however is also less uniform. Immersion involves submerging the specimen and produces a more uniform etching, however it is also easier to either under or over etch.

For difficult to etch specimens, the etching rate can be enhanced with temperature, ultrasonics, electrolytic or microwave energy.

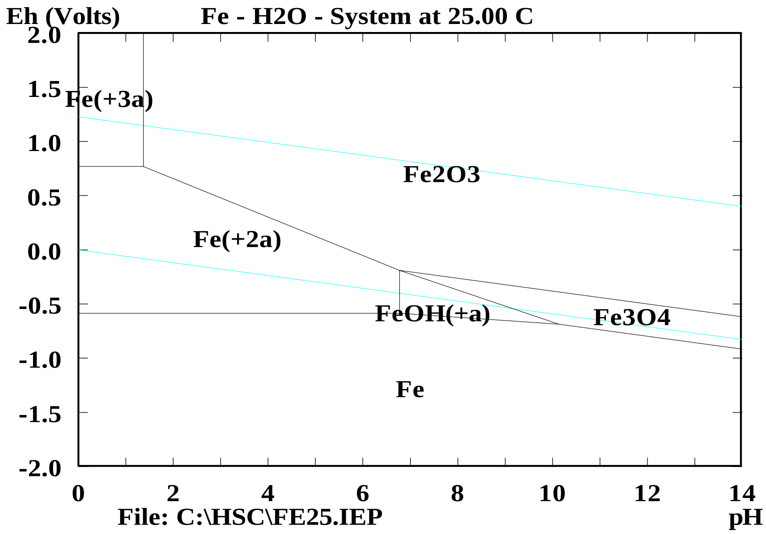
Inside this issue:

Considerations for chemical etching

- Chemical concentration
- Etchant temperature
- Agitation
- Specimen surface preparation



Pourbaix diagram for aluminum-water system.



Pourbaix diagram for iron-water system.

Pourbaix Diagrams

Pourbaix diagrams are thermodynamic diagrams which show the ability of metals to be attacked, dissolved or corroded at various pH and oxidation conditions. Although useful, they are limited because they do not address the kinetics of dissolution, which is often determined by experimental analysis or previous experience.

The upper left diagram is the Pourbaix diagram for the aluminum-water system. This diagram shows that aluminum will be attacked at pH values below 4 and at pH values above 10. From etching experiments high pH values attack the aluminum at a faster rate than at lower pH values.

Likewise, the Pourbaix for the iron-water system is shown in the lower left diagram. As indicated iron forms iron oxides at higher pH values forming iron oxides and therefore etching at higher pH values does not produce the best etching conditions. Also the iron dissolves to form either a Fe+3 or a Fe+2 species depending upon the oxidation potential. Note the most common etchant for iron and steels is nital which contains an oxidizing acid (nitric acid).

Common Chemical Etchants

Etchant	Composition	Conc.	Conditions	Comments
Kellers Etch	Distilled water HNO3 HCl HF	190 ml 5 ml 3 ml 2 ml	10-30 second immersion. Use only fresh etchant	
Kroll's Reagent	Distilled water HNO3 HF	92 ml 6 ml 2 ml	15 seconds	
Nital	Ethanol HNO3	100 ml 1-10 ml	Seconds to minutes	
Kallings Reagent	Distilled water Copper chloride (CuCl2) HCl Ethanol or methanol	40 ml 2 grams 40 ml 40-80 ml	Immerse or swab for few seconds to a few minutes	
Lepito's Reagent	Acetic acid Nitric acid	50 ml 50 ml	Swab	
Marble's Reagent	Distilled Water HCl Copper sulfate (CuSO4)	50 ml 50 ml 10 grams	Immersion or swab, etch for a few seconds	
Murakami Reagent	Distilled Water K3Fe(CN)8 NaOH or KOH	100 ml 10 grams 10 grams	Immerse or swab for seconds to minutes	Use fresh
Picral	Ethanol Picric acid	100 ml 2-4 grams	Seconds to minutes	Do not let etchant crystallize or dry - **explosive**
Vilella's Reagent	Glycerol HNO3 HCl	45 ml 15 ml 30 ml	Seconds to minutes	

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