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Cleaning chert with HF -a note

Christopher Maurer
Department of Anthropology
University of Florida
Gainesville, FL 32611
U.S.A.

Carriveau (1977, 1979) has suggested cleaning quartz grains in concentrated HF prior to measurement of their TL spectra. While this procedure has proven successful with quartz grains from pottery, caution should be used in extending it to chert.

During the recent dating of a heat-altered Florida chert tool, a sample of the tool was viced to $< 63\mu\text{m}$ and etched in 50% HF for 15 minutes. On addition of the acid, the chert immediately foamed into an opaque sludge with marked evolution of heat. Testing of the sludge showed that its TL signal had been destroyed. A repeat test showed that the foaming reaction was rapid, going to completion in 10-15 seconds. Weaker HF solutions were then tried. Tests of 1% (60 seconds and 10 minutes) and 2% (10 and 20 minutes) solutions showed neither caused foaming or heat evolution. Examination of the etched grains with a petrographic microscope confirmed that the 50% HF etched grains were completely destroyed while the 1% or 2% etched grains appeared unaffected.

This exothermic sludge may be similar to the "spectacular" failure observed by Huxtable (1982) in a flint from the La Cotte (France) site when she tested it with 40% HF. In both cases, there is the possibility of calcite contamination. Calcite alone, though, would not fully account for the effects observed as the calcite-HF reaction does not markedly evolve heat.

A clue to a possible origin of the effect may lie in a report by Donna Ruhl (Florida State Museum, pers. comm.) that a similar severe foaming and evolution to heat was observed in several North Florida clay samples when she added 40% HF to them. Her solution was to treat the clay samples three times with 10% HCl, with centrifugation and decantation of the supernatant plus a deionized water rinse after each addition, before using the HF. With our chert samples, we use a 3 minute, 38% HCl bath followed by a 20 minute, 4% HF etch.

In conclusion, chert/flint samples which foam when etched with 50% HF may be successfully etched in weaker HF solutions (1-4%) with or without a prior HCl bath.

References

- Carriveau, G. (1977) Cleaning quartz grains. Ancient TL, 1, 6.
- Carriveau, G. (1979) A comment on cleaning quartz crystals. Ancient TL, 9, 16
- Huxtable, J. (1982) Fine grain thermoluminescence (TL) techniques applied to flint dating. PACT J., 6, 346-352.

Reviewers Comments (S. Sutton)

The advantage of etching ground chert is unclear. As pointed out by Fleming, the purpose in etching quartz grains (from pottery, for example) is twofold; first, to increase transparency by removing material contaminated by impurity diffusion during weathering and, second, to remove part of the alpha-irradiated material. Chert "grains" are not obviously analogous. These "grains" would be expected to have more or less uniform transparency since they are composed of microcrystalline quartz and their surfaces have experienced no weathering. Also, the uranium and thorium distribution in the chert and in the "grains" is likely to be more uniform. It is stated that "grains" etched in dilute HF "appeared unaffected" under petrographic examination. How then was it determined that they were "successfully etched?"

Author's reply

Because chert frequently contains both a glassy phase and trace mineral impurities as well as quartz grains (Clark and Purdy, 'Electron microprobe analysis of weathered Florida chert' American Antiquity 44(3):517-524), the HF etch, preceded by a HCl etch, is used to try to remove the former while leaving most of the quartz grains behind. Secondly, "successfully etched" was imprecisely worded. The intended meaning was "did not foam into sludge".