

SAMPLE PRESERVATION AND HANDLING

INTRODUCTION

The importance of proper sample preservation and handling in the process of accurate materials analyses cannot be overemphasized. If samples are not cared for properly, important information may be destroyed, adulterated, or obscured. At the very least, improper handling introduces a measure of uncertainty into the analytical results. Thus, a few general principles are presented here to help preserve samples and the often critical data they contain. Please note that the principles presented here are not all encompassing. If you are uncertain how to best preserve and transfer a sample, contact the analytical laboratory directly for advice and instructions.

COMMANDMENTS OF SAMPLE PRESERVATION AND HANDLING

Don't Touch. Avoid touching the sample or area of interest with bare hands. Fingers inherently have significant amounts of organic and inorganic compounds that can contaminate the sample. Additionally, your fingers may pick up foreign material and transfer it to the sample or remove important deposits from the sample surface.

If you must use your hands to handle small samples, wear gloves. However, even gloves may transfer a certain amount of foreign material. Use clean tweezers or other handling tools for small samples.

As a rule of thumb, keep handling to a minimum, including poking, prodding, or scratching with tools or instruments. Such equipment may contaminate or destroy important material. Fracture surfaces are especially prone to physical damage that can inhibit accurate analysis. Simply touching mating fracture surfaces back together after a failure will destroy microscopic fracture features that may be the key to a conclusive determination of the fracture mode.

Choose samples wisely. Select samples for analysis that are representative of what you are trying to determine, i.e., typical contamination or typical material. In many cases, a control sample of "normal" material or components may be very useful as a comparison with failed or problem components. In some cases, several specimens may need to be submitted to determine commonalities or a range of conditions.

Preserve sample integrity. Obtain samples in a way that does not influence the measurements to be made. If a sample must be cut or removed from a larger piece, care must be taken not to contaminate or alter the area of interest. For example, the heat generated by flame cutting a metal sample may alter its microstructure and mechanical properties. Scraping on a hard surface with a metal instrument can produce wear debris from the instrument which is added to the component surface or to the collected surface deposits.

Submit a sample of appropriate size. Thermal analysis may require only a few milligrams. Quantitative chemical analysis may require a large surface area of several square millimeters or a few grams of material. If in doubt as to appropriate sample size, contact the analytical laboratory.

Preserve sample. Special sample handling and storage are often required to prevent potential changes in sample morphology and/or composition between time of sampling and analysis. Oxidation, evaporation, thermal degradation, or chemical interaction may occur if samples are not properly preserved.

Store samples in clean containers. This normally means new containers or those known to have been properly cleaned. Even if a previously used container appears clean, it may contain microscopic particles or liquids which could contaminate your sample and introduce uncertainty in the analytical results. If a sample must be shipped, package in such a way as to limit contamination or physical damage.

Avoid tape. Do not wrap samples or small particles in tape. Tape may leave an adhesive residue or remove critical sample constituents. Tape residue can create significant interference and uncertainty, particularly for analysis of organic compounds.

Identify and label. Clearly mark the sample containers to identify the contents. The source of the sample and, if applicable, its location within the source component should be recorded. Indicate the area of interest with a diagram rather than marking on the sample if possible. Data from the best preserved samples are meaningless if the sample and area of interest are not properly identified.

Obtain background data. Include significant background information about the sample and good instructions to the analyst with the sample. Provide a clear mandate for the analysis goals, i.e. explain why is the analysis requested. Background information about the sample that may help the analyst includes: where did it come from, what is it used for, and what has it been exposed to.

Provide control samples when possible. Submit a reference or control material(s) with the sample. A control sample will give you a baseline for comparison. If you are attempting to identify an unknown contamination, submit suspected sources of contamination along with the unknown for comparison. If an unusual condition is to be evaluated, comparison with a “normal” sample can be very useful.

Contact the analytical laboratory for specific sample preservation, handling, and shipping recommendations.

SPECIFIC GUIDELINES FOR HANDLING FRACTURES

Fractures, even those of hard or high strength metals, are fragile and subject to mechanical and environmental damage that can destroy important microstructural features. Thus, fractures must be handled with great care from sampling through analysis.

First, a fracture surface should be preserved as soon as possible following the failure to prevent environmental attack, such as corrosion or oxidation. Ideally, the fracture and surrounding surfaces should be dried with air and stored in a dry environment. At a minimum, the fracture area should loosely covered to protect it from rain or incidental physical damage. The fracture should not be sealed in an air tight container where water could condense and corrode the fresh fracture surface. If adequate protection or storage in a dry environment is not possible, the fracture may be coated with an oil, grease, or other material that will protect the surface, but not chemically attack it. (Do not coat surface if corrosion appears to a factor in the failure.) The coating used should be easily and completely removable for the subsequent analysis.

Do not try to fit two fracture halves together or pick at fracture surface, as this will mechanically damage critical surface features. If the fracture must be removed from a larger part, make the cut far away from the fracture site. Package so as to prevent any contact with the fracture surfaces. Small bumps and dings can significantly affect the fracture morphology. Wrap each component of the failure separately.