The Colorado Petrified Wood Sourcing Project

by

Kevin Black, Colorado Historical Society
Stephen Kalasz, Centennial Archaeology, Inc.
Frederick J. Fraikor, Colorado School of Mines
E. Craig Simmons, Colorado School of Mines

Paper presented at the 6th biennial meeting of the Rocky Mountain Anthropological Conference, Estes Park, Colorado, September 20, 2003

Abstract

A cooperative project is well underway between geologists from the Colorado School of Mines (CSM) and archaeologists representing the Colorado Council of Professional Archaeologists (CCPA) , to collect and analyze petrified wood samples from sites in the mountains and plains of Colorado. This pilot program is in part based on promising results from an earlier study conducted by a CSM student. The objective is to conduct geochemical trace element and microscopic cell structure analyses on both cultural (debitage) and non-cultural (natural) specimens, to evaluate both the chemical variability of agatized wood outcrops and the feasibility of using geochemical signatures to identify the provenance of petrified wood artifacts. If successful, this technique could help archaeologists identify the geographical source of petrified wood used to make flakes and tools in much the same way as is currently done with obsidian. This report will present data on the sampled localities, SEM microscopic and ICP–AES trace element results to date, and plans for additional research.

Introduction

Last year, the Colorado State Historical Fund awarded a grant to the Colorado School of Mines (CSM) and the Colorado Council of Professional Archaeologists (CCPA) for a petrified wood sourcing project. Microstructural and geochemical analyses are being conducted both to characterize petrified wood (PW) source materials and, hopefully, to match those geological signatures with artifacts from well-defined archaeological contexts.

At CSM, the research professor conducting the microstructure investigation of PW is Dr. Frederick Fraikor in the Department of Metallurgical and Materials Science Engineering. Dr Fraikor is Director of the Colorado Advanced Materials Institute (CAMI), a consortium of industry, state government and research universities in Colorado. Dr. Hans Joachim Kleebe, Associate Professor in the Department of Metallurgical and Materials Science Engineering, and several students are assisting in this phase of the project. For the geochemical analysis, Dr. E. Craig Simmons of the Department of Chemistry & Geochemistry leads the project. These investigators have recently conducted other archaeological materials and geochemistry research projects involving chemical analyses of petrified wood artifacts in Colorado and obsidian artifacts from South America. Working on these projects, student Janine Rowsey (2001) completed her Master's thesis two years ago in Materials Science, and another is working on her Master's degree in Geochemistry.

The CCPA's role on this project is to provide archaeological expertise, support for field investigations, and help with access to collections and raw material sources. Thus far, archaeologists Bridget Ambler, Kevin Black, Robert Brunswig, Ed Friedman, Ken Frye, Stephen Kalasz, Frank Rupp, Christy Smith, Mark Stiger, Susan Struthers, Gordon Tucker, and Meg Van Ness have contributed their time in procuring samples for analysis; help in sample collection also has come from avocational archaeologists Marvin Goad and John Ross, and rancher Jim Moore.

Purpose of the Grant

It is well known that, in portions of Colorado, one of the most common raw materials used by early inhabitants was petrified (silicified) wood. For example, at the Magic Mountain site near Golden, petrified wood tools and flakes made up approximately 28% of all lithics excavated during 1994 and 1996 (Kalasz and Shields 1997), and much higher frequencies are typical on sites nearer the source of the petrified wood. Many sources of petrified wood throughout Colorado have been identified by archeologists (e.g., Black 2000), and undoubtedly there are many more yet to be discovered. As with obsidian studies (e.g., Shackley 1997, 1998), it would be quite helpful to be able to definitively correlate a specific petrified wood artifact with a specific source. This would provide us with a clear geographical path from its source as a raw material to its final site as a finished tool and, more important, illuminate early geographical movements and possible interregional relationships. Unfortunately, unlike some archeological artifacts such as *Olivella* sp. shell from the Gulf of California, petrified wood cannot be sourced visually or macroscopically. Instead, advanced analytical techniques must be developed to enable investigators to chemically trace an artifact made from fossilized wood.

Thus, a principal goal of this investigation is to chemically and microscopically analyze petrified wood artifacts from archaeological sites on the Eastern Plains, Gunnison Basin and North/Middle Park areas of Colorado to identify their original site source and possible geographical pathways. Those areas were selected in this pilot study because local source materials were known to exist, and additional localities in South Park and the San Luis Valley have been added due to the interest and knowledge of archaeologists with local expertise.

The first objective pursued thus far has been to investigate the feasibility of using Scanning Electron Microscopy (SEM) to use features of wood microstructure to possibly identify the species of the fossilized trees. Now underway is the pursuit of another objective, to adapt advanced analytical technologies at CSM, including Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP–AES), to geochemically correlate petrified wood artifacts with specific raw material source sites.

Educational outreach is an integral part of this project. All of the significant results of the investigation will be condensed into press releases and articles by the CSM Public Relations Department. Updates and articles are to be posted on the CSM and CCPA web pages. The project will also develop relevant displays at the museum on the CSM campus that can be loaned to other sites. In addition, the close working partnership between the CCPA and the School of Mines should induce new interdisciplinary technology transfer exchanges not only between professional archaeologists and faculty but also between their students and volunteers. The CSM geologists consider it vital to infuse their engineering students with an understanding of

Colorado history and culture, and similarly for traditional anthropology and archaeology students to understand and work with the latest breakthroughs on the cutting edge of technology.

The results of this project, then, will be of interest in addressing both archaeological and geological research questions. For example, geologists would like to know:

1. What is the actual process of silicification of the original wood?
2. How chemically variable are individual petrified wood sources? Are there chemical variations across log sections from bark to heartwood?
3. Can the plant species of fossil wood be determined using SEM?

For archaeologists (see Church 1994), relevant research questions include:

1. Is the prehistoric use of petrified wood sources highly localized? For example, is Palmer Divide petrified wood used only by bands occupying the Denver Basin?
2. What is the evidence that petrified wood from specific sources was traded? For example, does petrified wood from the Gunnison Basin appear in Front Range or southeastern Colorado contexts?
3. What is the evidence for long-distance travel vs. exchange or influence from adjoining regions?
4. How valid are the various transhumance models posited for the Front Range?
5. What are the geographic ranges of seasonal rounds?
6. Did lithic production and procurement strategies change after the introduction of metal and the horse?

No one involved in this project expects to answer all of these questions. Again, this is a pilot project and we expect that there will be subsequent efforts to pursue lines of inquiry raised by the results of this study.

Methods

Field Collection

The collection of specimens in the field began in July 2002 and the bulk of the field work now has been completed. Whether natural or cultural, the archaeological site or fossil locality was documented—usually by taking UTM coordinates with a Global Positioning System (GPS) receiver. Photographs were taken both of the local landscape (Figure 1) and of potential specimens still *in situ.* Sample bags were appropriately labeled and field notes were taken. In some cases, CSM geologists accompanied the archaeologists and were able to examine local bedrock outcrops to supplement information from geological maps for information on the host formations.

Lab Procedures, Microstructure

At CSM Dr. Hans Joachim Kleebe (Figure 2), Director of the Electron Microscopy Laboratory, is assisting the project team to provide additional expertise in preparing and analyzing petrified wood (PW) specimens by Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). Eladio Perez Lopez was a graduate student in the

Figure 1. Photo of hillside in South Park where samples labeled "Friedman #1" were collected from the outcropping petrified log fragments in the foreground.

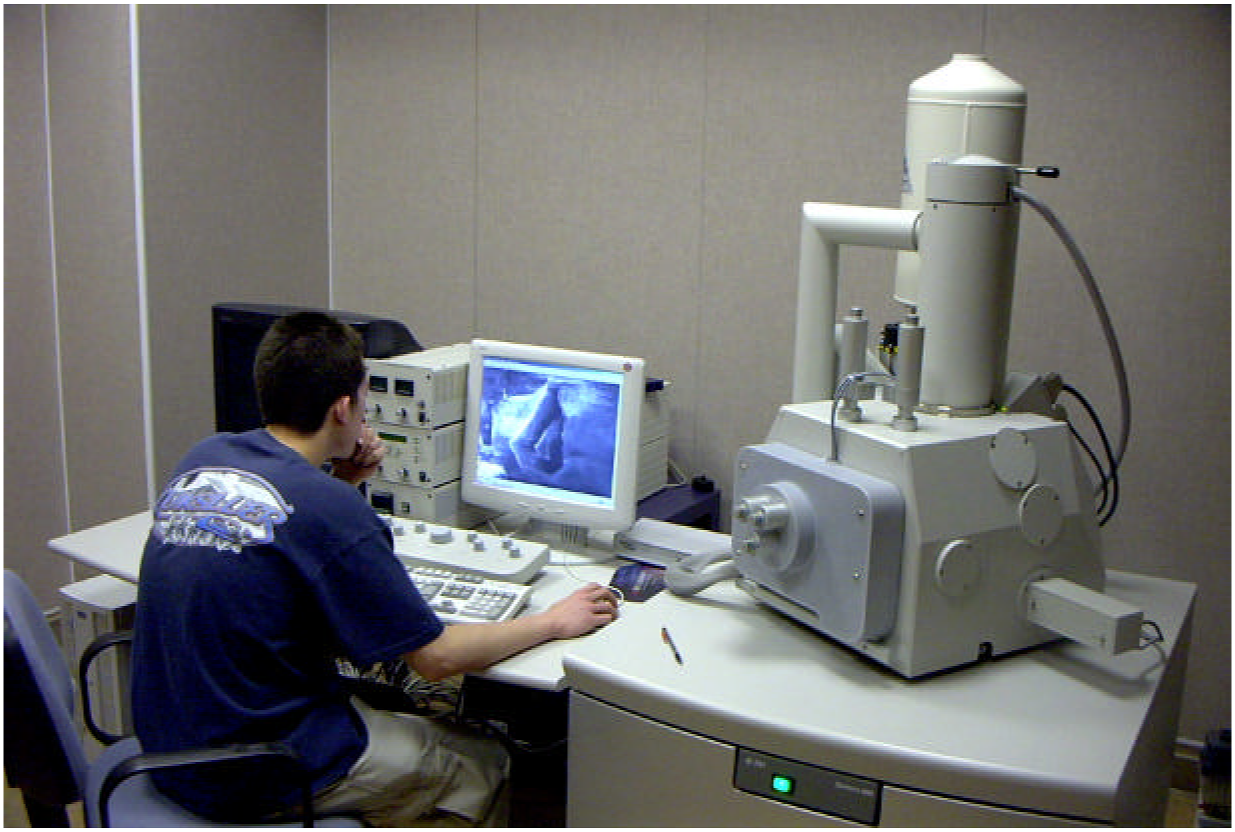
Figure 2. Photo of Dr. Kleebe at the TEM.

Metallurgical and Materials Engineering Department, and successfully developed specimen preparation techniques for the SEM. He has been awarded a fellowship to a Paris university and left the project in August 2002. He has been replaced by Tim Casias (Figure 3), a student in Metallurgical and Materials Engineering who is continuing the work (Casias et al. 2003). Derek Hudson, a work study honors student in computer science, is assisting Dr. Fraikor with library and research work.

The SEM provides magnification up to 50,000× and is the primary means of examining and defining the microstructure of prepared PW surfaces. By contrast, the TEM requires thin-section preparations and can examine samples at up to 5,000,000×. At that level of magnification, aspects of the atomic structure of PW can be discerned, but such detail is expected to be of lesser value in this project than larger structural data from the SEM.

In order not to use archaeological samples for the initial development of specimen preparation techniques and parameters for chemical and microstructure analyses, three pieces of petrified wood from Green Mountain (Jefferson County) were donated to the project from a private collection by a retired CSM mining engineer. The specimen preparation parameters are as follows.

Figure 3. Tim Casias, a CSM student, works at the SEM.



PW samples were first examined in the as-received field condition to study the surface structure (Figure 4), and for chemical analysis by Energy Dispersive X-ray Spectroscopy (EDS). For the Green Mountain material no discernable structure was observed in the weathered condition, and EDS analysis showed only silicon and a small amount of sodium as expected. SEM samples approximately 1.25 cm in length were cut from the documented PW specimen and carefully polished through succeeding grit sizes. These SEM samples were subsequently etched for 7–9 hours in a hydrofluoric acid solution to reveal the silicified wood microstructure. The SEM samples were mounted with carbon tape or glue on a sample holder, and gold-coated for examination at varying magnifications in the microscope. (The gold coating is necessary for insulating materials such as PW to minimize the charging and build-up of electrons on edges, which appear as washed out white areas.)

For TEM, the specimen preparation technique is a much longer and more difficult process, because a TEM specimen must be approximately 1000 ? or less in thickness for the 200 kw electron beam to penetrate through the atomic lattice of the minerals. Hence, 3 mm diameter samples were first cored from the PW sample. These were ground and polished below 200 µ with diamond paste, and the sample was dimpled with a 3 µ diamond solution to a thickness approximately 15 µ in the center section. Then, the disks were placed in an Ion Mill machine for the final thinning by bombardment with argon gas ions.

Figure 4. Photo of 5GA122 field samples before processing for microstructure analysis.

Lab Procedures, Geochemistry

Several CSM students have been, or will be, working on this phase of the project. A junior Chemistry major, Andrea Elliott, has been organizing, photographing and cataloguing the present collection of samples. She has also assisted in the selection of samples for analysis, and prepared them for chemical analysis. She will then learn to apply Inductively Coupled Plasma–Atomic Emission Spectroscopy (ICP–AES) technology, and will examine the variability of trace elements across samples of PW. Kerry Aggen, a graduate student working on her Master's degree thesis on sourcing obsidian artifacts in South America (Simmons et al. 2003), also is helping with literature surveys and microprobe work. Eleanor Steffi and Helen Kearney, graduate students in Chemistry and Materials Science, respectively, are assisting with the SEM correlation work, ICP–AES, and laser analyses.

After careful examination of the petrified wood samples with both megascopic and microscopic methods, representative pieces are pulverized to a fine powder. Weighed aliquots of this powder are then dissolved with a mixture of high-purity $HF–HClO_4–HNO_3$ acids, then diluted to a known volume. The abundances of at least 14 major and trace elements are determined for these solutions, enabling the calculation of the concentrations of these elements in the original PW. This phase of the project is just getting underway.

Current Status of the Project: Preliminary Results

Table 1 lists the locations where archaeological and/or fossil samples of PW have been collected thus far. To date, over 300 samples of PW from 39 Colorado localities (Figure 5) have been obtained and archived at the Colorado School of Mines. Collection has focused on well-documented PW source zones in the central third of Colorado, supplemented by materials from more distant areas less known as sources of PW. The project has benefited immensely from the contributions of geologists, archaeologists, and private citizens who were not involved in the initial development of the grant proposal.

While the current collection is more than adequate for analysis of natural source materials, a bit more work to obtain collections from a couple more archaeological sites may yet occur. For example, in July of this year, 11 samples from one softwood tree stump (*Sequoia affinis*, Figure 6) and one hardwood stump (*Chadronoxylom* sp.) were collected at Florissant Fossil Beds National Monument. Since the sequoia stump is a prominent artifact of the park, samples were only obtained from the debris at the base of the tree. Likewise, debris samples were also removed from around the hardwood stump for analysis. These specimens from Florissant are more scientifically definitive with respect to identification and origin than the sequoia sample from a private collection previously examined by SEM. The next step will be to obtain permission to examine archaeological artifacts that were collected in the Florissant area during the 1970s and are now housed in the museum at Rocky Mountain National Park.

Electron microscopy is continuing with microstructure analysis to see if species can be identified, beyond the more general recognition of hardwoods and softwoods. Energy Dispersive X-Ray Fluorescence (EDXRF) and Laser Ablation – Inductively Coupled Plasma Mass Spectroscopy (LA-ICPMS) have been used to ascertain sample heterogeneity and possible elemental composition variations in petrified wood. This has been followed by use of Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) on archaeological samples to provide a data base of trace elements, which will be statistically evaluated. All data then will be correlated with archaeological sources and sites, and a final report prepared.

Table 1. Geographical Coverage of Collections

SITE	LOCALITY	# OF SAMPLES COLLECTED	SAMPLE TYPE(S)
Coal Creek	Buckley	2	Paleontological
5AH416	Parker	7	Archaeological
5BL55	Front Range	3	Archaeological
5CH192	East-central plains	2	Archaeological
5DA515	Highlands Ranch	2	Archaeological
5DA1008	Castle Rock	1	Archaeological
5DA1658	Parker	2	Archaeological
5DA1659	Parker	2	Archaeological
5DA1681	Parker	1	Archaeological
5DA1765	Parker	3	Archaeological
Kiowa #1	Parker	16	Paleontological
Moore Ranch	Parker	21	Archaeological and Paleontological
Parker	Parker	15	Paleontological
5EL157	Bijou Basin	1	Archaeological
5EL166	Bijou Basin	2	Archaeological
5EL168	Bijou Basin	1	Archaeological
5EL588	Bijou Basin	1	Archaeological
5EL612	Bijou Basin	1	Archaeological
F. Rupp #1	Middle Park	12	Paleontological
Antelope Pass	Middle Park	5	Paleontological
Culbreath	Middle Park	5	Paleontological
F. Rupp #4	Middle Park	14	Paleontological
5GA122	Middle Park	6	Paleontological
5GA639	Middle Park	1	Paleontological
5GA680	Middle Park	17	Paleontological
5GA2002	Front Range	2	Archaeological
5GA2262	Front Range	1	Archaeological
5GA2712	Front Range	2	Archaeological
5GA2788	Rabbit Ears Pass	19	Archaeological and Paleontological
Smith Creek	Rabbit Ears Range	11	Paleontological
Stiger #1	Gunnison Basin	41	Paleontological
Stiger #2	Gunnison Basin	30	Paleontological
5JA1377	North Park	2	Archaeological
Green Mountain	Front Range	16	Paleontological
Magic Mountain	Front Range	22	Archaeological
5KW103	East-central plains	1	Archaeological
5KW105	East-central plains	1	Archaeological
5LR10246	Front Range	1	Archaeological

Florissant #1	South Park	3	Paleontological
Florissant #2	South Park	8	Paleontological
Friedman #1	South Park	16	Paleontological
SpArP-2001-24	South Park	5	Archaeological and Paleontological
SpArP-2001-25	South Park	12	Archaeological and Paleontological
5PA2697	South Park	1	Archaeological
5SH1999	Trickle Mountain	5	Paleontological?
5SH2000	Little La Garita Creek	3	Paleontological?
5SH2001	English Valley	3	Paleontological?
ICPW-001	Pawnee Buttes	2	Archaeological
RMPW	Pawnee Buttes	1	Archaeological
WSPW-001	Pawnee Buttes	1	Archaeological
WSPW-002	Pawnee Buttes	3	Archaeological

Figure 5. Colorado map depicting sample collection sites.

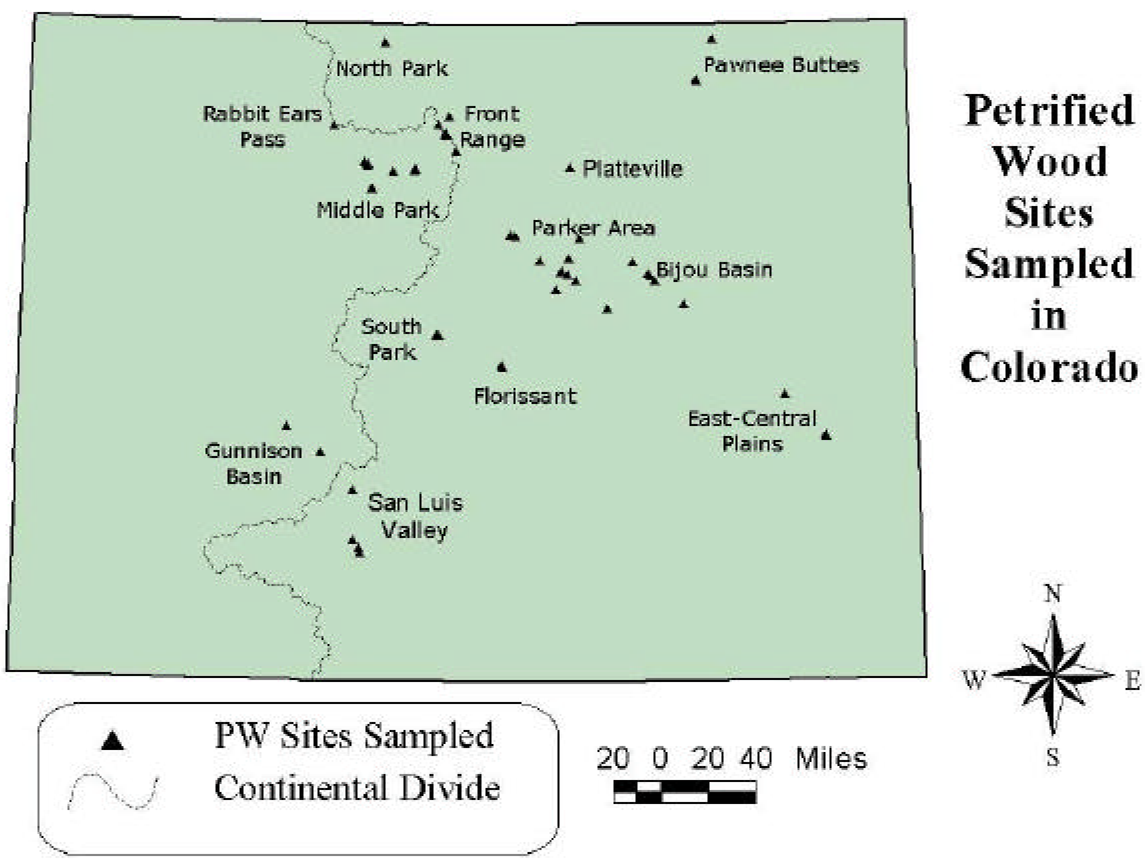


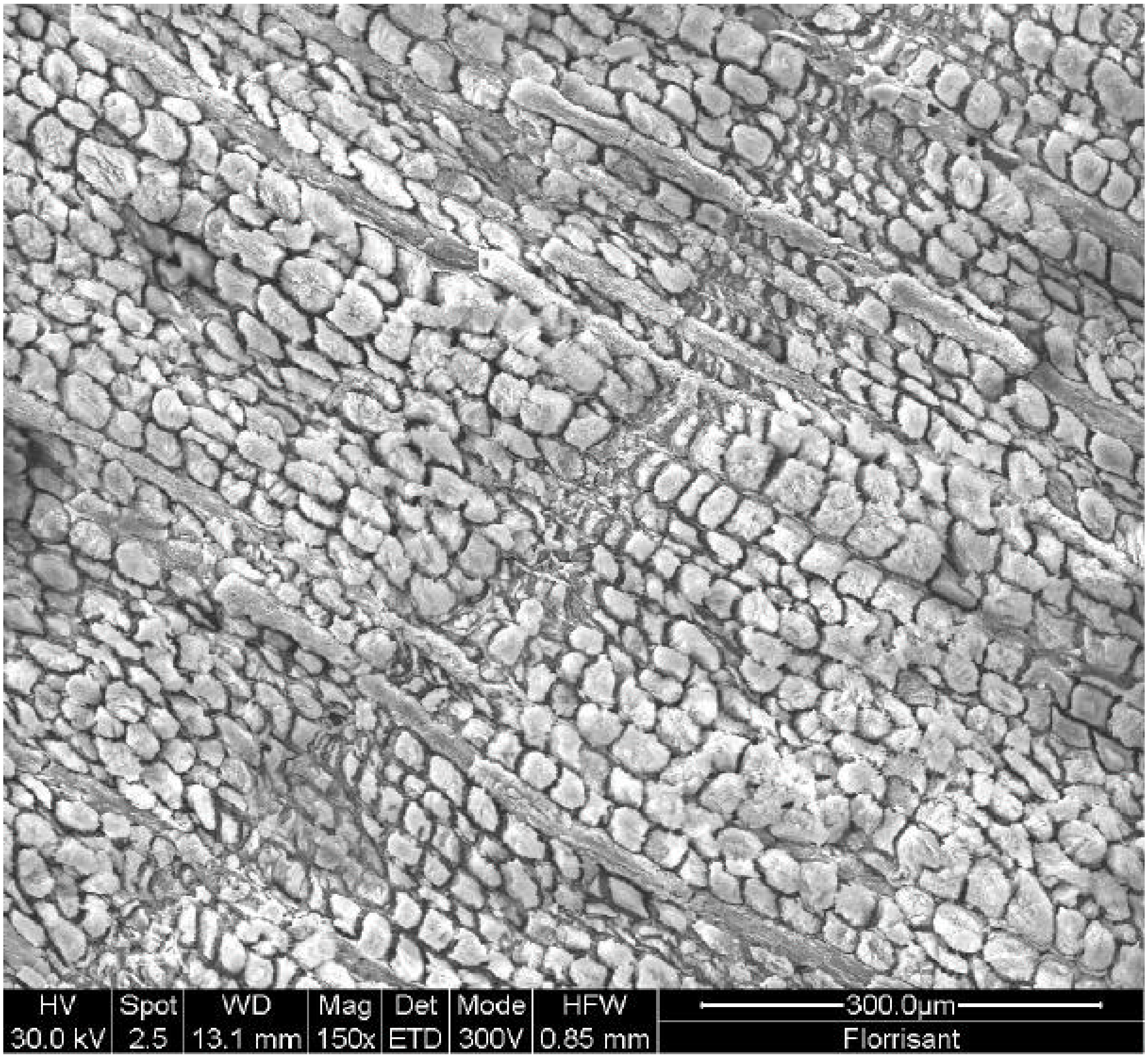
Figure 6. Chuck Harvell photo of sequoia stump at Florissant Fossil Beds National Monument.

Microstructure

Figure 7 is a photomicrograph of a PW sample from a source near Florissant, CO. The cross section (end view) of this particular sample at 150× clearly delineates a uniform structure of rows of small cells throughout the specimen with no evidence of larger tubular vessels, possibly indicative of a softwood such as sequoia or pine. This structure is comparable to other local Florissant PW identified as species *Sequoia affinis.* According to the literature on wood analysis, a hardwood such as oak or maple should have a different morphology of mixed large and small cells (Figure 8). Samples from the Smith Creek area north of Middle Park show such a hardwood structure , unlike the majority of other samples collected thus far.

It has become clear that there is a limit to the utility of such analyses at higher magnifications. The following images show a sequence of PW samples at increasing levels of detail. Figure 9—a sample from the Gunnison area seen at 50×—while showing some cell structure, is not magnified enough to show sufficient detail. The next sample (Figure 10) from Highlands Ranch magnified 100× is more informative, as is the same Gunnison sample (Figure 11) at 700×. However, the Gunnison sample at 1500× (Figure 12) is at a little too high a level of magnification. Details of individual PW cells can be discerned, but not the overall structure of cellular arrangements. Geologically and paleontologically, such cell details may be of interest as

Figure 7. Sample Florissant #9, SEM ×150, showing its probable softwood structure.

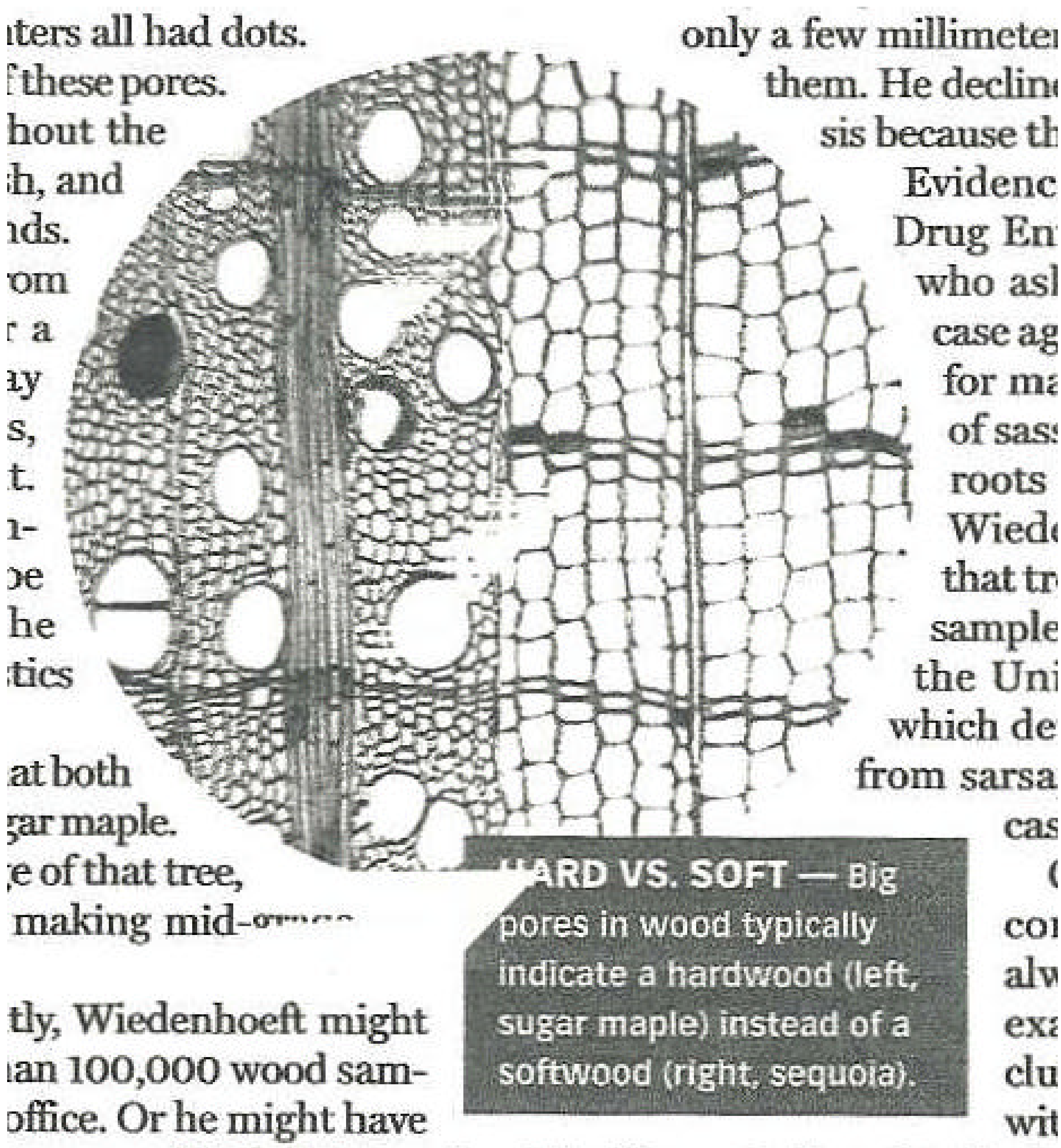


in a close-up (Figure 13) from a Gunnison sample at 20,000× that is possibly an iron pyrite crystal. Thus, magnifications above about 2000× are less likely to produce results useful in characterizing PW materials in ways that meet our archaeological sourcing objectives.

One other aspect of this lab work is the difficulty in preparing the samples for the SEM. In order to best view the PW microstructure, the sample must be cut directly across the grain of the original wood. While the wood grain is obvious in many of the natural source materials, many suspected PW artifacts are so highly silicified that the wood grain is either difficult to see or entirely replaced.

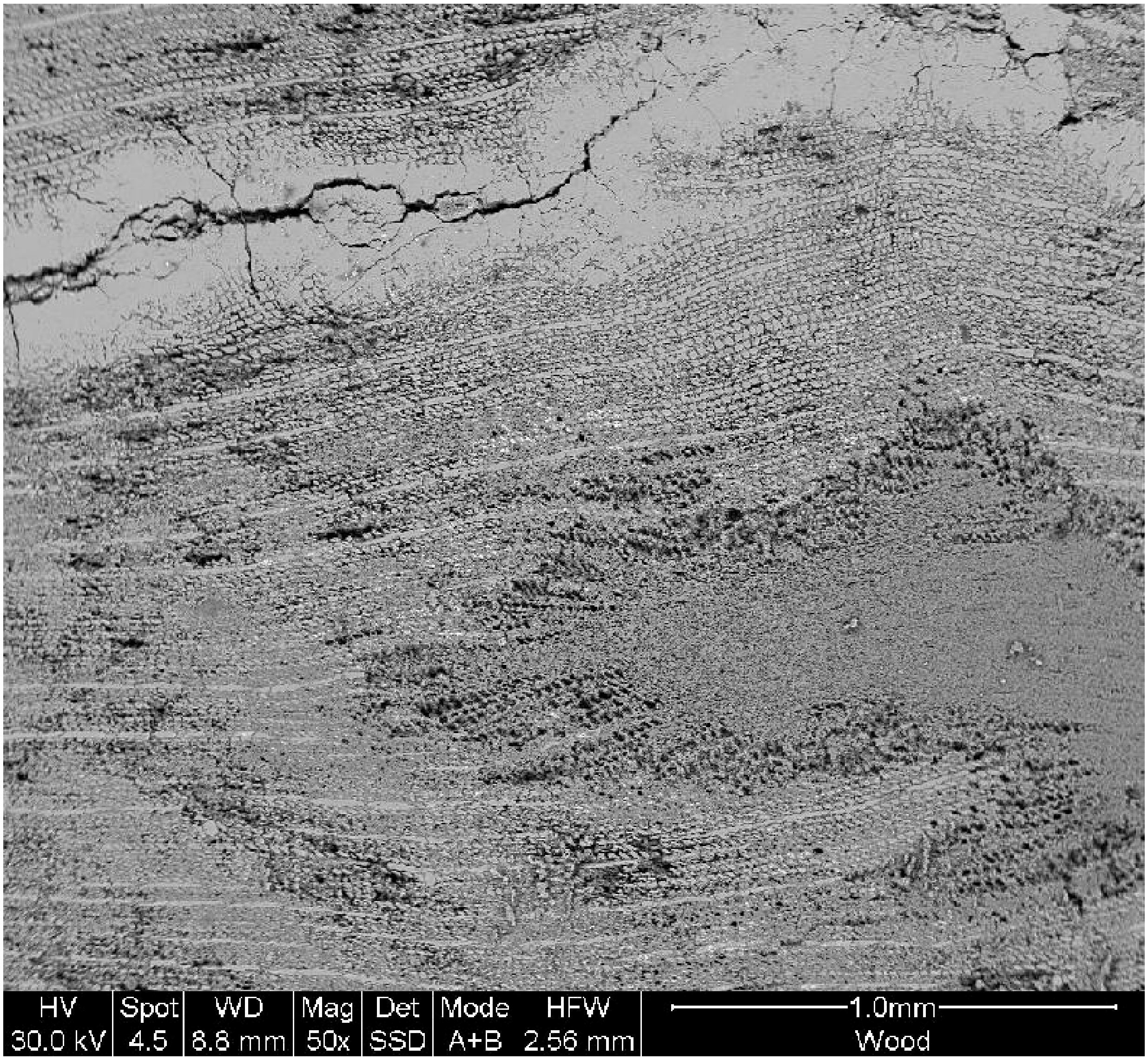
As with the chemical analyses, the heterogeneity of silicified wood often makes specimen preparation difficult and the search for any signs of a retained wood structure an even lengthier one. The process of determining the duration of etching an SEM sample l cm long and only 2 or 3 mm thick in the hydrofluoric/ammonium fluoride solution becomes a "trial and error"

Figure 8. Structural distinctions between hardwood and softwood.



experiment and more of an art than a precise science. While an etch of eight hours may work for many samples, the same duration for the initial two hardwood samples from Florissant completely dissolved the specimens. Conversely, insufficient etching results in a low-relief sample that does not show much detail under the SEM, even after eight hours of etching such as with a sample from North Park (Figure 14). Gold coating the SEM samples is still another variable since a gold film that is too thick will mask some details of the microstructure of the silicified wood while one that is too thin will allow a buildup of electrons on the surface introducing unwanted "highlighted" spots.

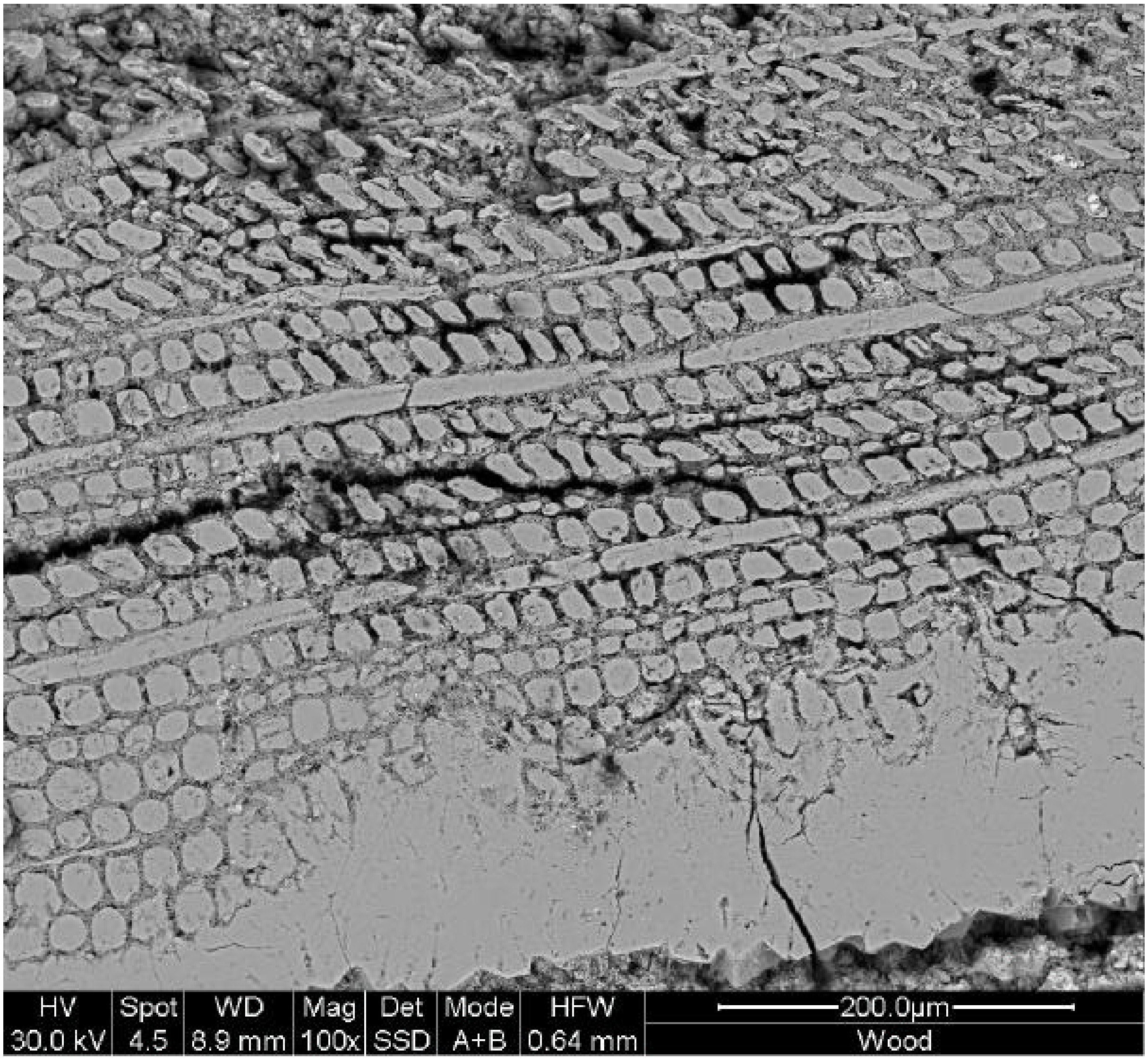
Figure 9. Photomicrograph of sample Stiger #1 from Gunnison, SEM ×50



Also, factors such as compression from geological process and/or location of the specimen in the original tree can distort the retained microstructure and inhibit identification from a small artifact. Figure 15 shows a well-defined band of retained cells and rays from the sequoia stump debris at Florissant that transition into a convoluted, twisting microstructure on both sides of the band. A similar twisting is seen in the colliding intersection of compacted cells in Figure 16 from 5DA1681 in the Parker area.

As a general observation it appears that denser, more "agatized" PW that would be favored by flintknappers for various kinds of tools is also more difficult to etch and observe definitive retained microstructures. Nonetheless, twenty-one samples of PW from the eastern plains, Front Range, and mountain park regions of Colorado have been successfully polished and etched to identify the petrified wood as a hardwood or softwood species examined using the two scanning

Figure 10. Photomicrograph of Highlands Ranch sample #1, SEM ×100.

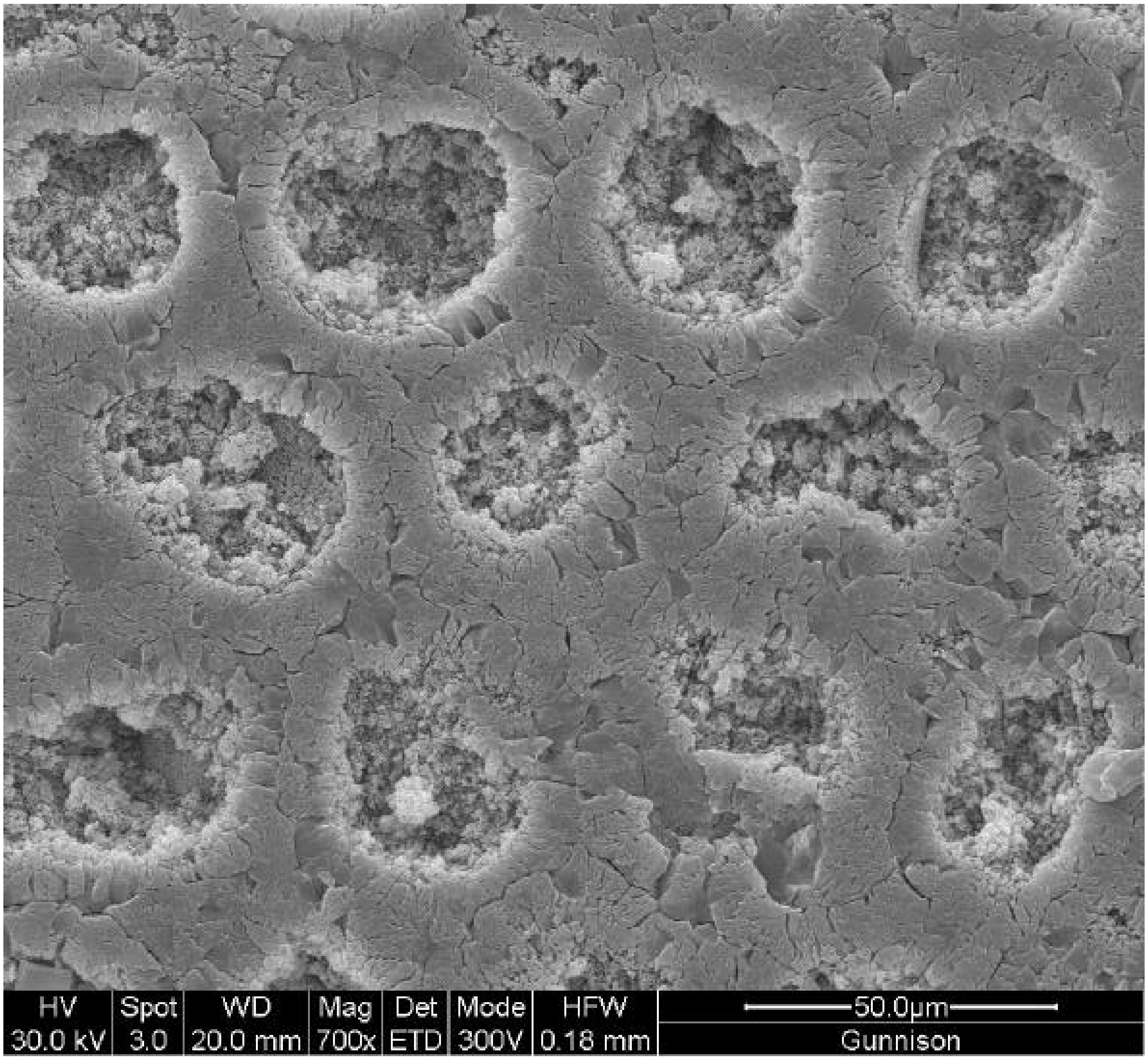


electron microscopes at CSM. The vast majority examined thus far exhibit softwood morphology.

Geochemistry

As previously noted, the geochemistry phase of the project using EDXRF, LA–ICPMS, and ICP–AES is in its early stages and, thus, there are only a few results to report at this time. The main potential problem with sourcing silicified wood is the degree of significant heterogeneity of elemental distribution in a sample. Composition variations may be introduced across the diameter and length of a fossilized log, dependent on a number of variables including varying diffusion rates and geological compaction processes, the specific environmental conditions, and the composition of the surrounding soil. This heterogeneity potential poses several questions for the graduate student's experimental approach:

Figure 11. Photomicrograph, Stiger #1 SEM ×700



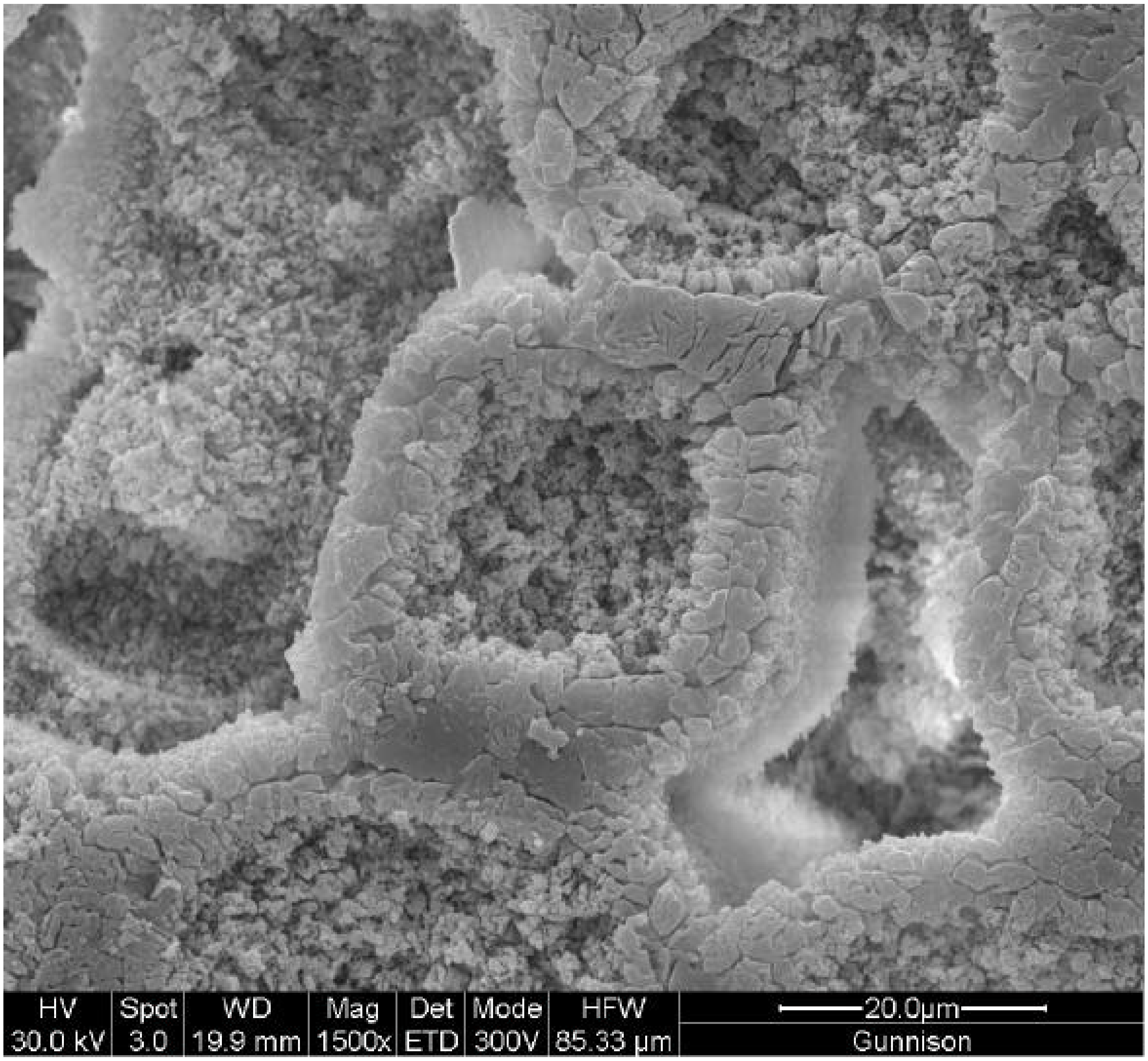
Qualitatively, what will be the statistically acceptable differences among means for the presence or absence of specific elements in the elemental distribution (test) results?

Quantitatively, what will be the statistically acceptable differences in concentrations for elements observed to be present in the semi-quantitative results?

What will be the statistically acceptable differences for the distribution of elements within a sample of petrified wood from one source, between samples within the same source, and between samples from different sources and sites?

With these questions in mind, a small petrified wood "log" 33 cm in length and 8-12 cm in diameter collected by the project team from the Moore Ranch in Parker was selected as a good specimen to analyze for chemical heterogeneity (Figure 17). This log was found on the surface a

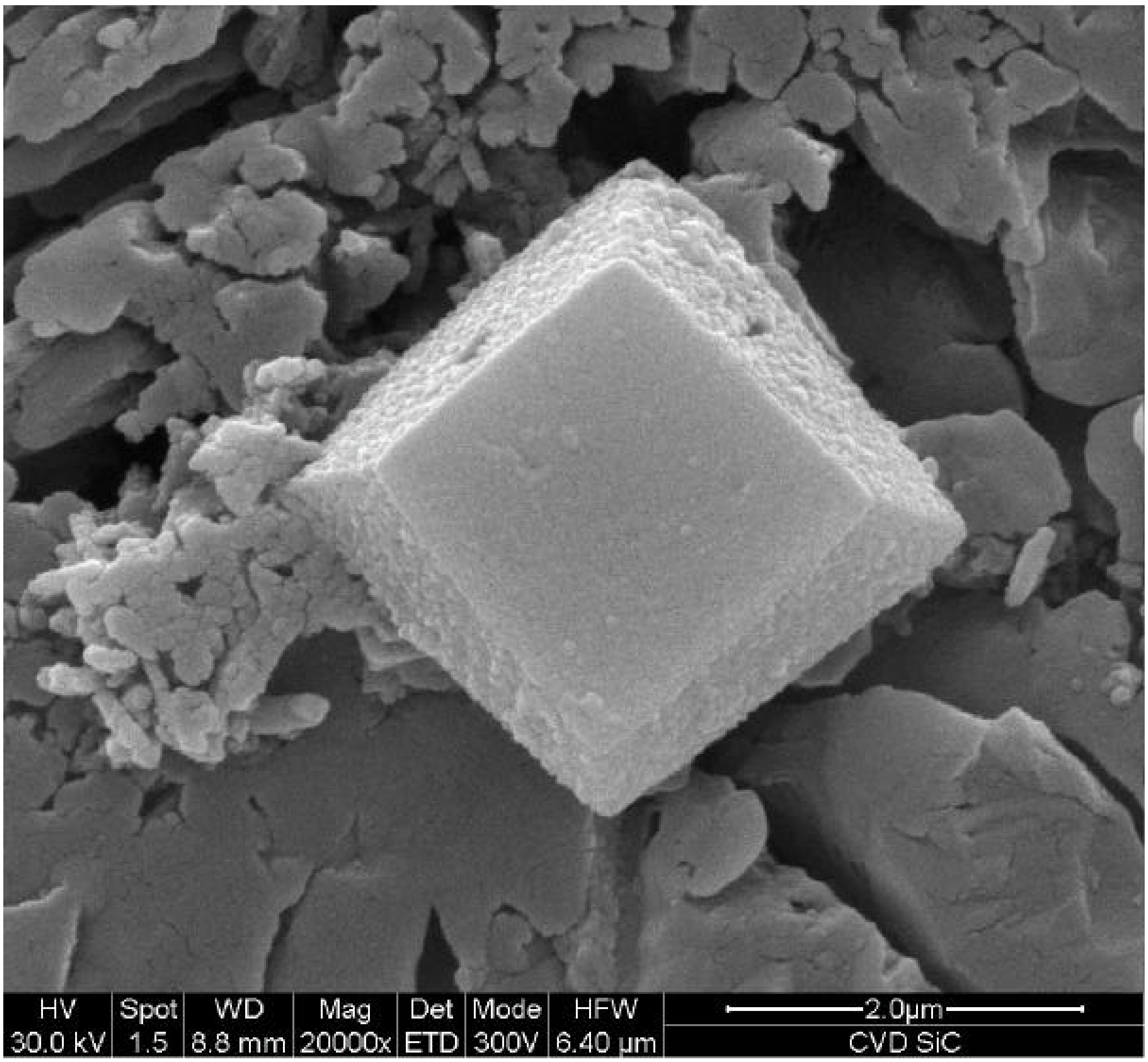
Figure 12. Photomicrograph, Stiger #1 sample from Gunnison, SEM ×1500.



few hundred yards west of a giant 23 m-long PW log preserved by Mr. Moore (Figure 18). Although the ranch is not recorded as an archaeological site, there is significant evidence of worked debris on the ranch grounds showing that this area—like many others in the Parker region—was a source of PW raw material for local hunter-gatherer groups. Hence, this specimen was a good representation of source material as well as being a sample of PW large enough for heterogeneity measurements.

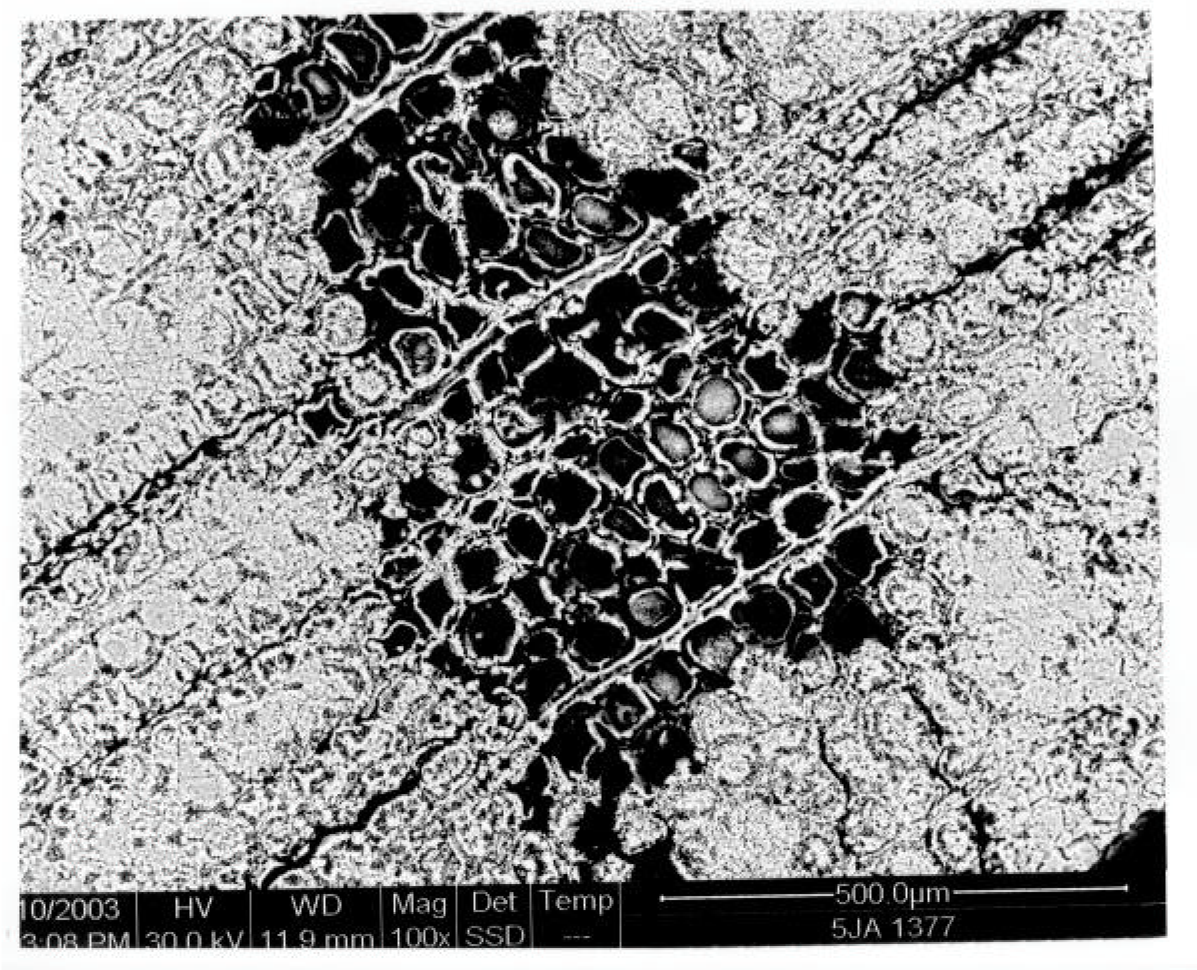
The specimen was sliced into 2-3 cm diameter cross-sections using a diamond saw lubricated with water. Analyses were performed with no etching or polishing of the cut faces to eliminate possible contamination from preparation materials. Preliminary semi-quantitative Energy Dispersive X-ray Fluorescence (EDXRF) scans first were run on the face of the cut cross-sections using an Oxford Instrument EDXRF, and using a geological materials analysis protocol written by Hazen laboratory. These results provided background elemental composition data on PW and show that, in addition to the expected major constituents of SiO_2 and Fe_20_3, other main elements were detected such as Zr, Al_2O_3, Ba, and CaO. A second scan of another quadrant showed significant variations in composition in the raw data.

Figure 13. Photomicrograph of sample Stiger #1 from Gunnison, SEM ×20,000.



Sections of the log were then scanned using Laser Ablation–Inductively Coupled Plasma Mass Spectroscopy (LA-ICPMS) equipment at the USGS laboratories in Lakewood. Four quadrants of the "face" surfaces of the cross-sections were scanned without polishing or etching. Standard number NIST_610 were first analyzed using LA-ICPMS as a calibration standard to compare the spectrum for that homogeneous material with the spectrum from the petrified wood specimen (Figure 19). As was expected, the heterogeneous petrified wood displayed a considerable variation of chemical constituents in this preliminary experiment.

Figure 14. Photomicrograph of 5JA1377 softwood (backscatter mode), SEM ×100



A specimen of PW collected from Florissant Fossil Beds National Monument (Sample #FSS 9, hardwood log debris) was then examined. The resulting spectrum (Figure 20) indicates that the Florissant specimen contains significantly more barium than the sample of petrified wood from the Moore Ranch at Parker. Again, these are initial results subject to subsequent statistical analyses. These heterogeneity measurements will be followed by chemical analyses of all the PW specimens using Inductively Coupled Plasma–Atomic Emission Spectroscopy at Colorado School of Mines. Obviously, the distinctiveness in trace element signatures of PW sources is of great interest to archaeologists, and will have a major bearing on the outcome of this pilot study. More complete results on this critical part of the project will be available soon.

What's Next?

Conclusions

The final report on this pilot study is due at the end of this year, but hopefully the project is a beginning, not the end. Think of obsidian studies, in which a long period of research was necessary to establish baseline data for the kind of routine uses archaeologists have come to expect. It will be no different with petrified wood, even if our results exceed expectations. Ancient uses of petrified wood were not limited to flintknapping (Figure 21), yet progress in flaked stone research alone will require many more projects beyond this one.

Finally, endless thanks are expressed to the archaeologists here today and all the others who have contributed their time and samples to this effort. We couldn't do it without you.

Figure 15. Photomicrograph of distorted and compressed cells along edges (eight hour etch), Florissant sample FSS-3, SEM ×100

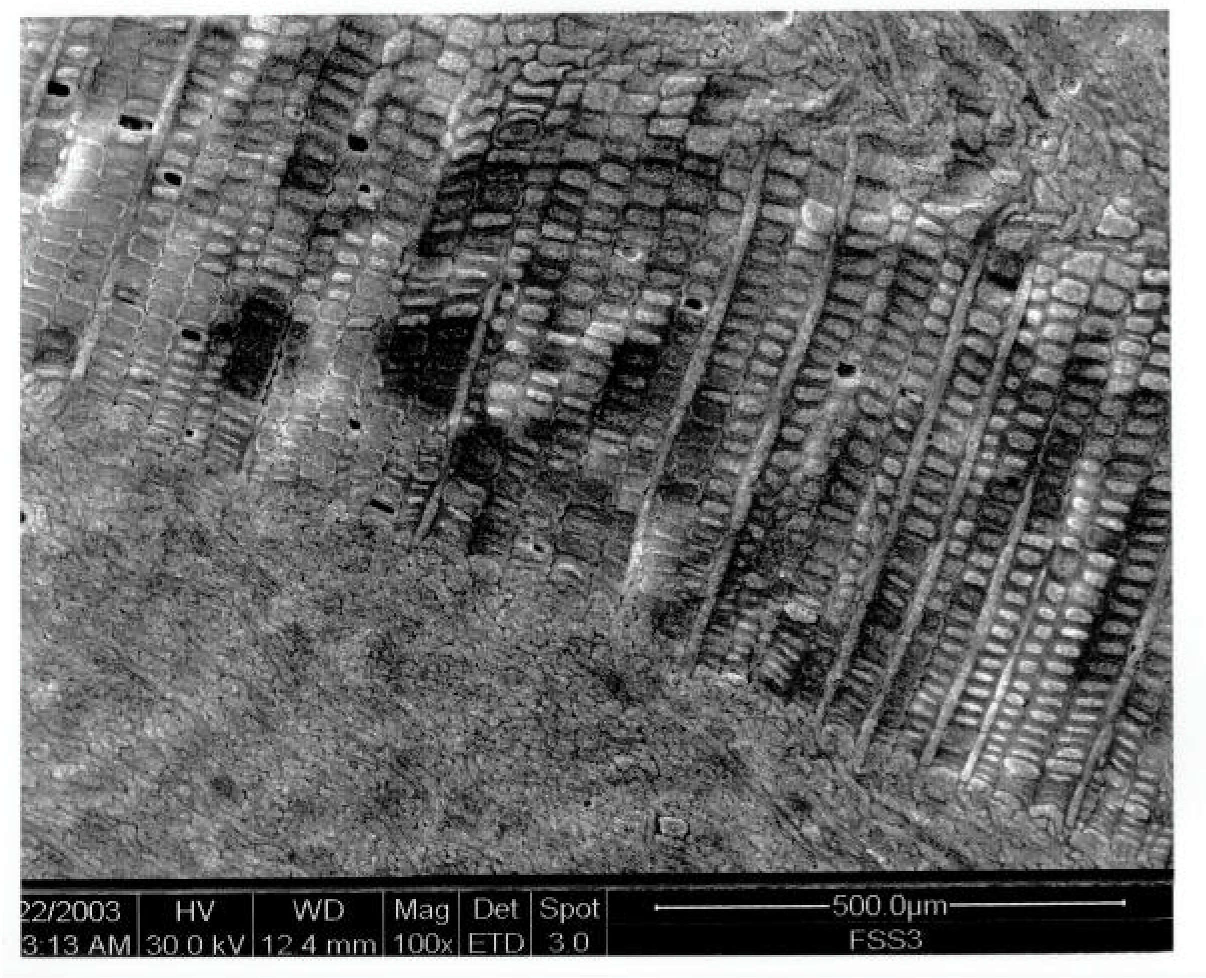


Figure 16. Photomicrograph of compressed cells in a V-shaped microstructure (eight hour etch), from site 5DA1681 in the Parker area, SEM ×100.

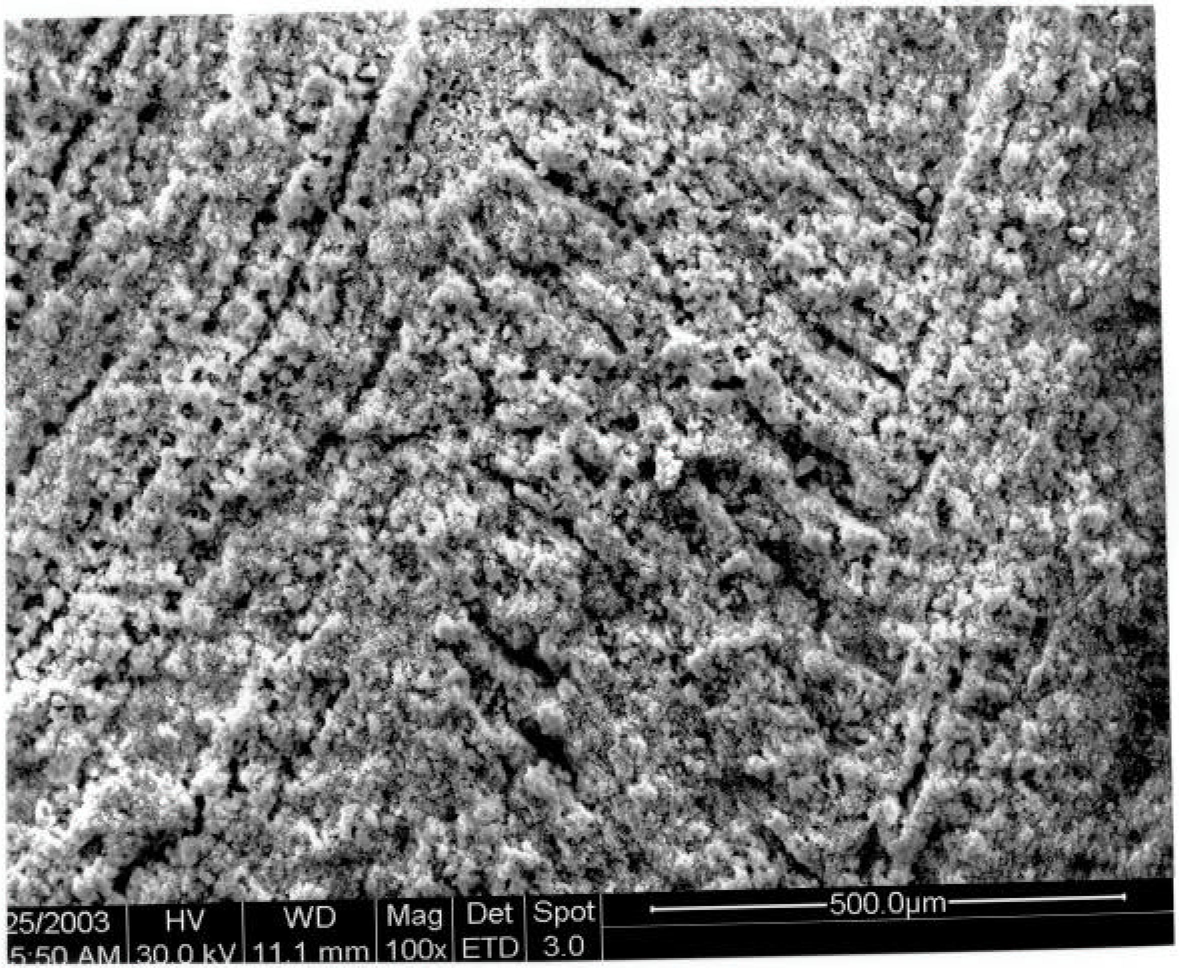


Figure 17. Dr. Fred Fraikor holding PW log segment from the Moore Ranch.

Figure 18. Huge petrified wood log preserved on the Moore Ranch near Parker.

Figure 19. Graph of the trace element spectrum for the K2 (Moore Ranch) sample.

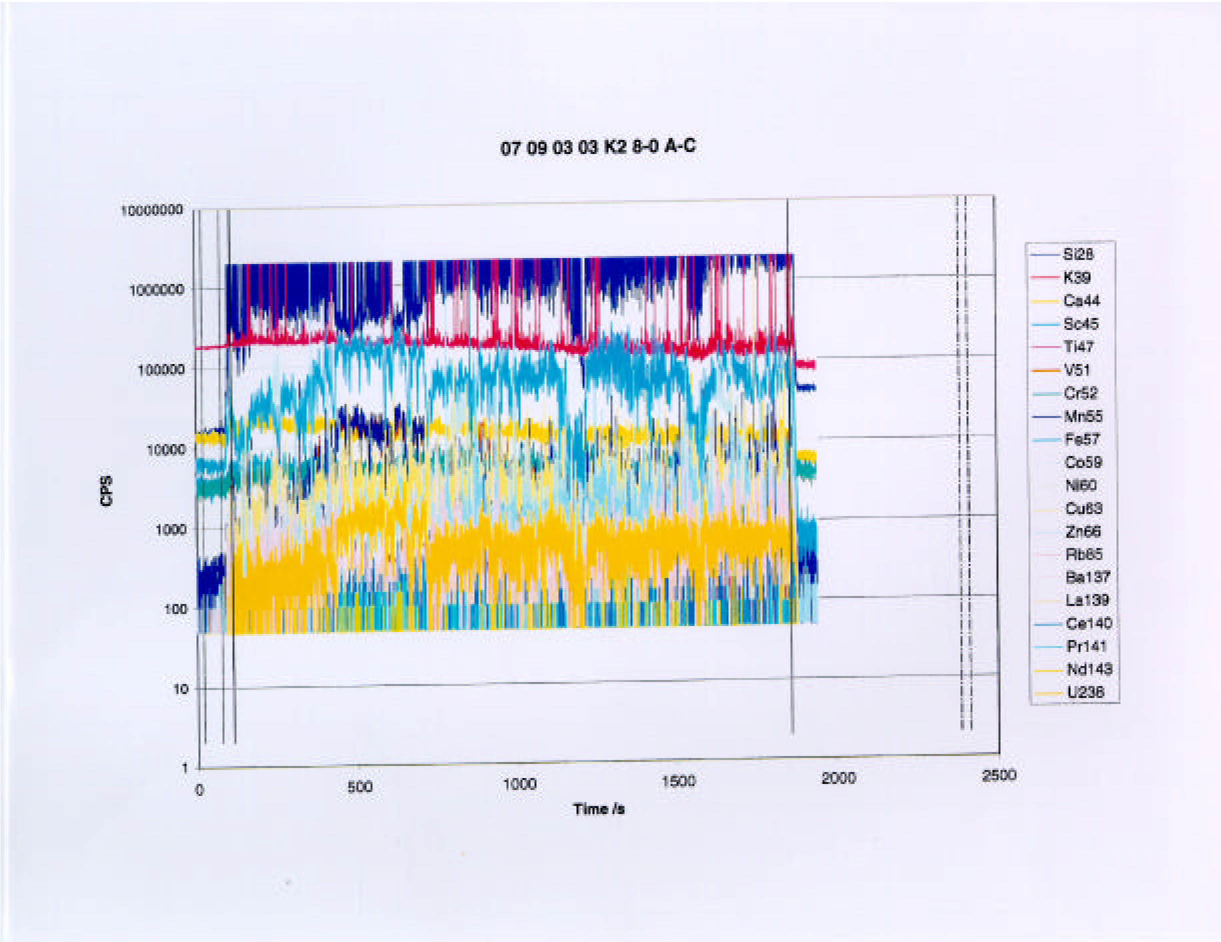


Figure 20. Graph of the trace element spectrum for the FSS-9 (Florissant) sample.

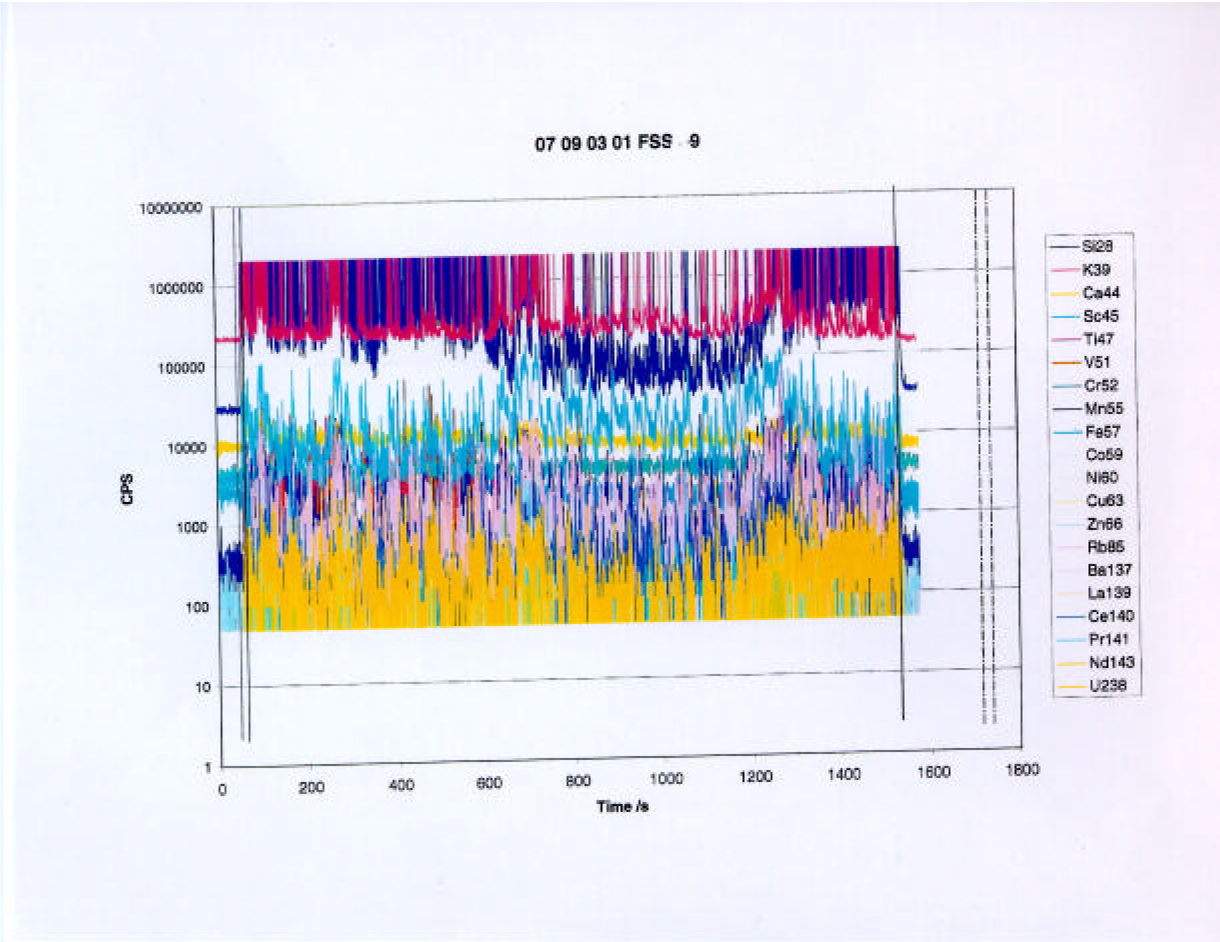


Figure 21. Agate House at Petrified Forest National Monument.

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