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A Preliminary Characterization of "Libby-Type Amphiboles" by SAED (Selected Area Electron Diffraction)

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Both AHERA and Yamate TEM methods for analyzing airborne asbestos samples mandate the identification of asbestos fibers based on the morphological, chemical and structural characteristics. It is logical to expect that the same guidelines should also be applied to the TEM analysis of Libby-type amphiboles.

Although a substantial amount of EDX (energy dispersive X-ray) spectroscopy analysis has been performed on amphiboles from Libby, Montana, I am not aware of any published literature on the characterization of Libby-type amphiboles by SAED analysis. Since EPA is in the process of developing guidelines to define Libby-type amphiboles, I think that the emphasis should be placed not solely on the chemical composition or elemental profiles by EDX. Such guidelines should also include the structural properties by SAED even though not every TEM analyst is proficient in this technique, which is indispensable to TEM asbestos identification.

This report is a brief summary of our preliminary results on the SAED characterization of one regular and three Libby-type amphiboles provided by USGS through CDM.

CELL PARAMETERS OF LIBBY-TYPE AMPHIBOLES

Table 1 summarizes the cell parameters of two winchite specimens from Libby, one richterite plus a tremolite and an actinolite from JCPDS database. The cell parameters of Libby amphiboles are very close to those of actinolite and tremolite.

Table 1. Cell parameters of winchite, richterite, tremolite and actinolite (Å)

Mineral	a(Å)	b(Å)	c(Å)	β(°)	Source
Winchite-1	9.885	18.032	5.288	104.54	Wylie and Verkouteren (2000)
Winchite-2	9.861	18.003	5.276	104.37	Wylie and Verkouteren (2000)
Richterite	10.03	18.415	5.234	104.97	webmineral.com
Tremolite	9.84	18.02	5.27	104.9	JCPDS 13-437
Actinolite	9.860	18.110	5.340	105.00	JCPDS 19-1601

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ZONE-AXIS SAED PATTERNS OF LIBBY-TYPE AMPHIBOLES

Zone-axis SAED patterns were obtained from all four samples at diffraction camera constant 22.9 mm•Å using a single-tilt sample holder. Because the predominant platy morphology elongated along c-axis exhibited by all fibers (Figure 1), [201] and [021] zone-axes occur fairly frequently. One zone-axis pattern for each sample was selected and presented in Figures 2 to 5. The indexing results of these SAED patterns are summarized in Table 2.

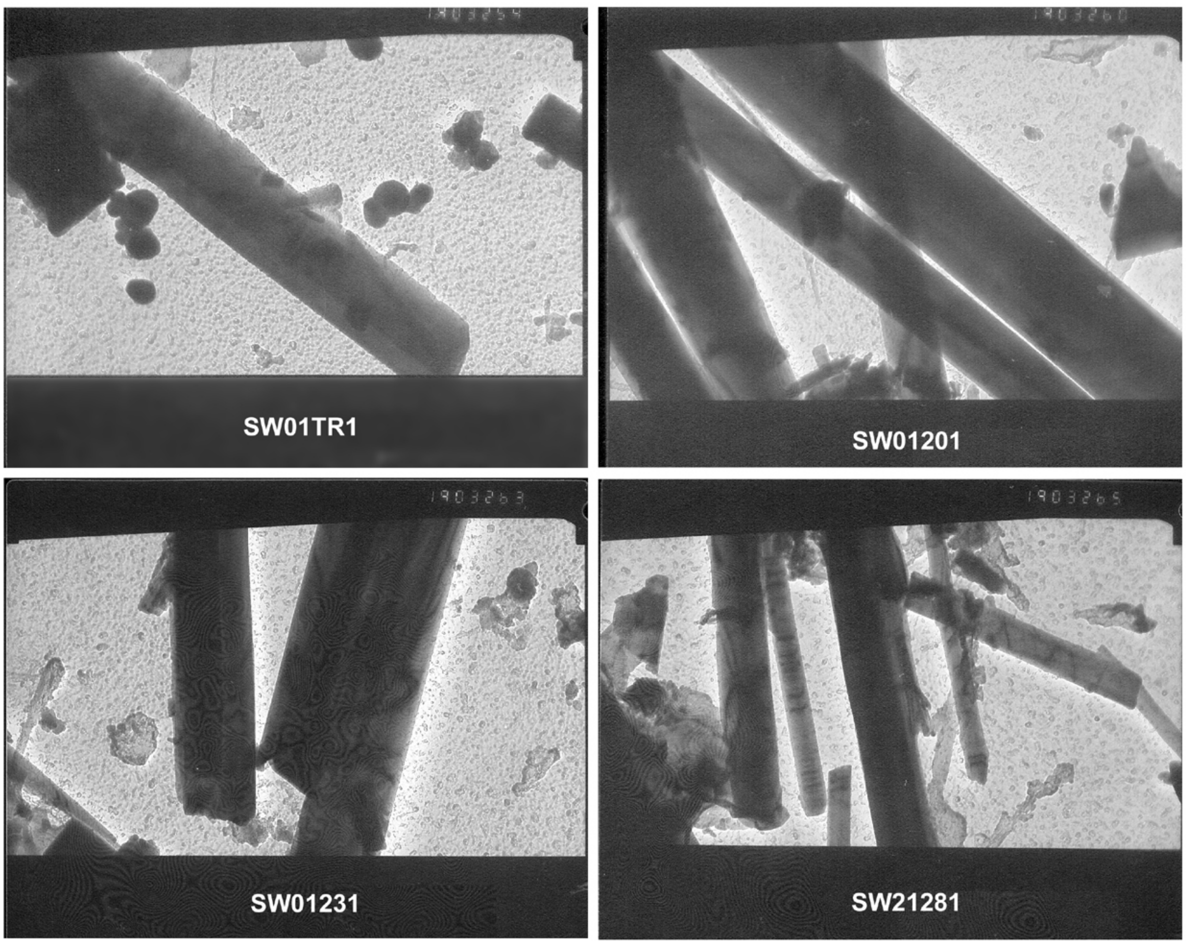

Figure 1. Micrographs of one regular tremolite (SW01TR1) and three Libby amphiboles from USGS. Visible Newton rings in the two micrographs at the bottom were resulted from interference at the interface of scanner glass and negatives. All micrographs were taken at 19,000X magnification.

Table 2. Indexing results of zone-axis patterns of four USGS amphiboles

Mineral	d-spacing (Å)			(hk0)^(hkl)	Zone-axis	Remarks
	(020)	(200)	(11,-2)	Φ(°)		
SW01TR1		4.638	2.617	89.5	[021]	Measured from Fig. 2
SW01201		4.610	2.694	89.8	[021]	Measured from Fig. 3
SW01231	9.160		2.642	81.5	[201]	Measured from Fig. 4
SW21281	9.245		2.655	81.5	[201]	Measured from Fig. 5
Winchite-1		4.770	2.613	89.0	[021]	Calculated from cell parameters in Table 1
	9.016		2.613	81.7	[201]	
Winchite-2		4.776	2.610	88.9	[021]	Calculated from cell parameters in Table 1
	9.002		2.610	81.7	[201]	
Richterite		4.845	2.591	89.8	[021]	Calculated from cell parameters in Table 1
	9.208		2.591	81.9	[201]	
Tremolite		4.753	2.607	89.4	[021]	Calculated from cell parameters in Table 1
	9.010		2.607	81.7	[201]	
Actinolite		4.762	2.641	89.3	[021]	Calculated from cell parameters in Table 1
	9.055		2.641	81.7	[201]	

Observations and Conclusions

1. The observed d-spacings of both (hk0) and (hkl) reciprocal lattice points (or direct crystallographic planes) of the four amphiboles are reasonably close to those of documented winchite, richterite, tremolite and actinolite. This quantitative structural information confirmed that the fibrous minerals not only exhibited compositional profiles that are characteristic of amphibole series but also have cell parameters characteristic of these solid solution series as well. According to AHERA and Yamate protocols, positive identification of amphibole asbestos can then be established.

2. It may take some time to find a zone-axis pattern especially when the sample holder is the single-tilt type. However, all these amphiboles belong to C2/m space group and their c-axis lengths are in a narrow range (5.2Å to 5.4Å). They invariably form acicular or tabular morphology elongated along c-axis and rest on the filter surface with c-axis more or less perpendicular to the electron beam. Therefore, (h01), (h02), (0k1), (0k2), (hk1) and (hk2) are usually at high angles to the filter surface or at small angles to the electron beam. These lattice

planes often generate layer lines with spacing close to c-axis length (5.2Å to 5.4Å) or half of c-axis length (2.6Å to 2.7Å). A trained TEM analyst should be able quickly find such an orientation that exhibit these types of layer lines without having to search for a zone-axis pattern. And both AHERA and Yamate protocols consider the observation of these types of layer lines to be an acceptable confirmation of amphibole structures.

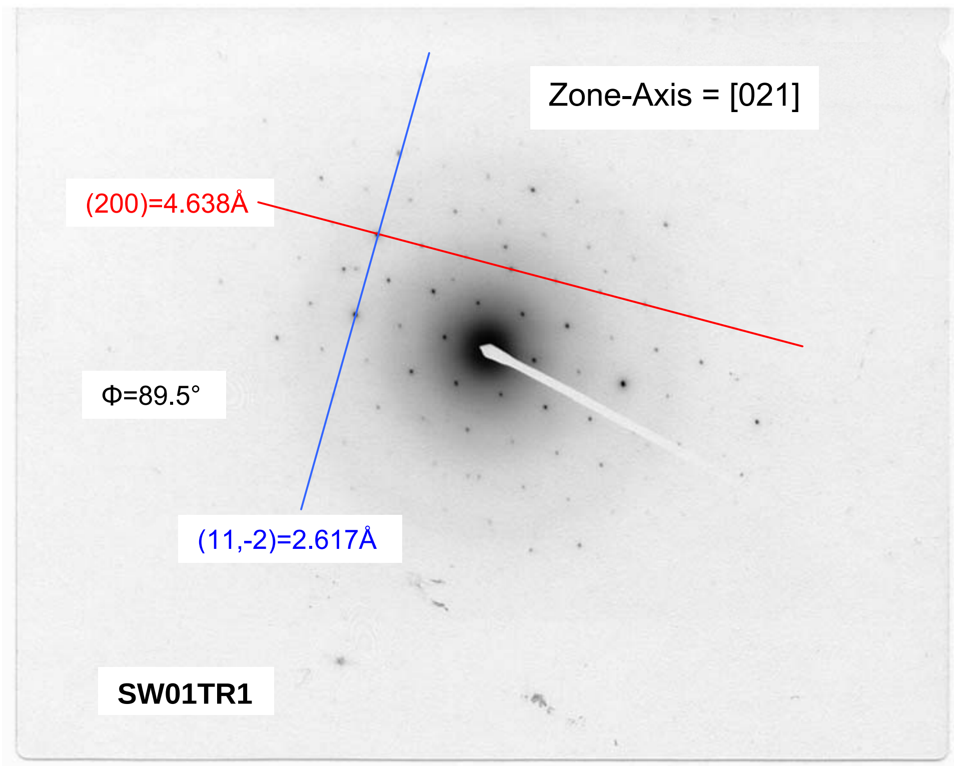

Figure 2. [021] zone-axis SAED pattern of SW01TR1. We identified this amphibole to be a "normal" tremolite based on the compositional profile by EDX. The layer lines parallel to the (hk0) diffractions (red line) are spaced at twice of (11,-2) or 5.23Å. *Remember: SAED patterns are images of reciprocal lattice and the d-spacing is inversely proportional to the geometric distance*. Even if we do not spend much time to look for a zone-axis pattern, this typical "5.2Å to 5.4Å layer lines", which is characteristic of asbestosform amphiboles, can be relatively easy observed without much searching. The objective aperture can be easily calibrated to provide a quick evaluation of the d-spacing of layer lines on the fluorescence screen.

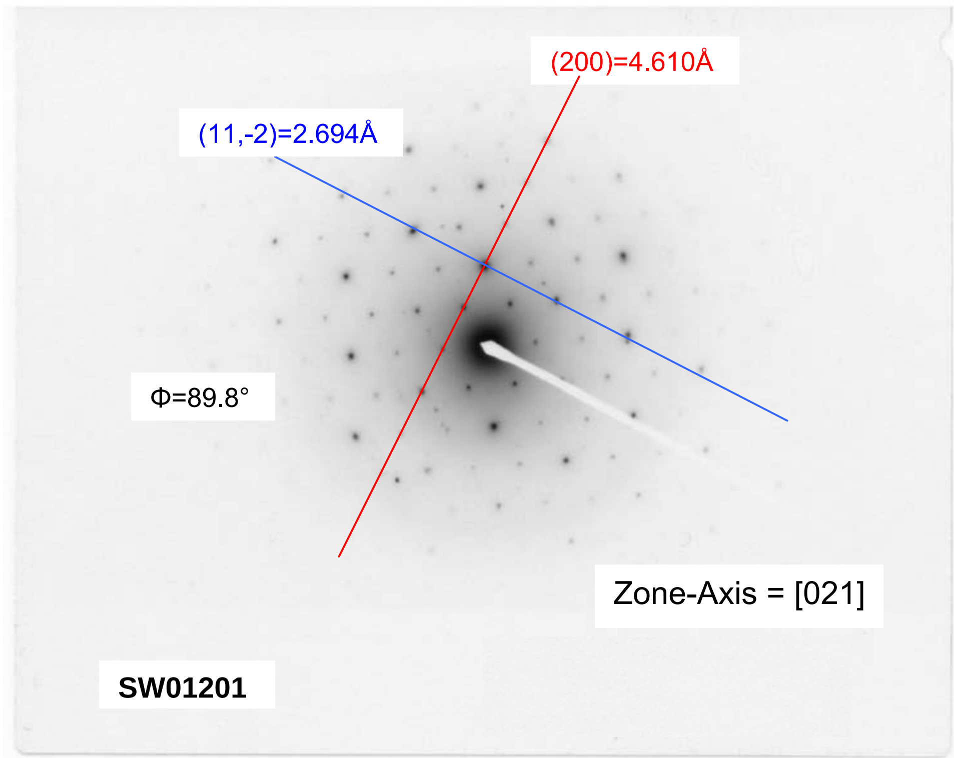

Figure 3. [021] zone-axis SAED pattern of SW01201. We identified this amphibole to be a Libby-type amphibole based on the compositional profile by EDX. The layer lines parallel to the (hk0) diffractions (red line) are spaced at twice of (11,-2) or 5.39Å. *Remember: SAED patterns are images of reciprocal lattice and the d-spacing is inversely proportional to the geometric distance*. Even if we do not spend much time to look for a zone-axis pattern, this typical "5.2Å to 5.4Å layer lines", which is characteristic of asbestosform amphiboles, can be relatively easy observed without much searching. The objective aperture can be easily calibrated to provide a quick evaluation of the d-spacing of layer lines on the fluorescence screen.

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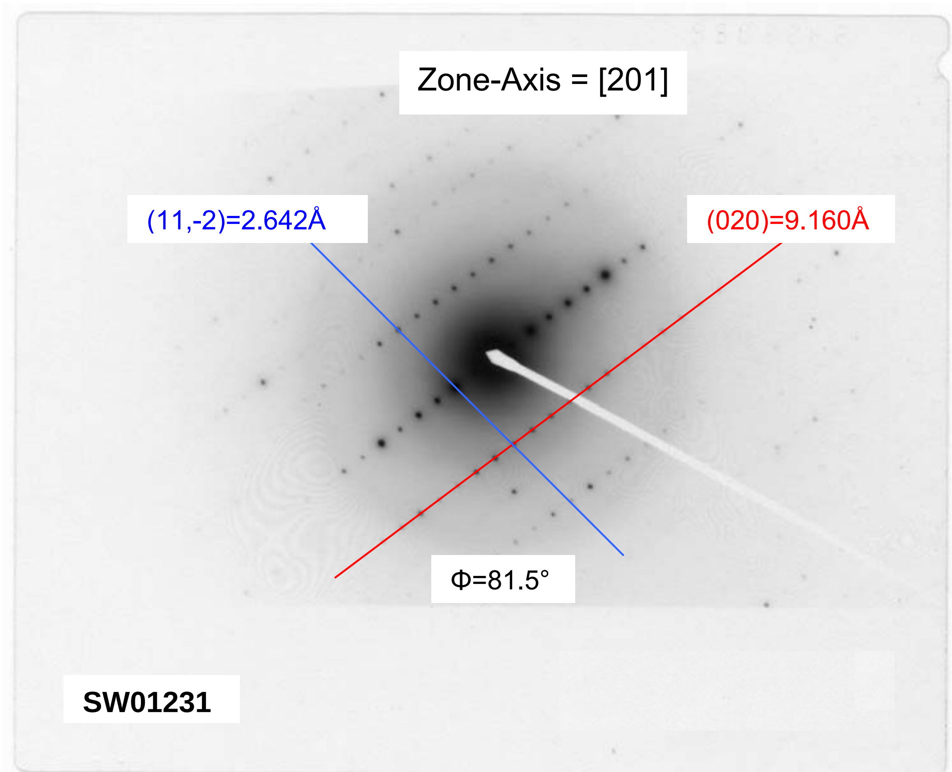

Figure 4. [201] zone-axis SAED pattern of SW01231. We identified this amphibole to be a Libby-type amphibole based on the compositional profile by EDX. The typical "5.2Å to 5.4Å layer lines" are absent at this orientation. However, the "2.6Å" layer lines are striking visible. Even if we do not spend much time to look for a zone-axis pattern, the typical "2.6Å layer lines", are characteristic of asbestosform amphiboles, can be relatively easy observed without much searching. The objective aperture can be easily calibrated to provide a quick evaluation of the d-spacing of layer lines on the fluorescence screen.

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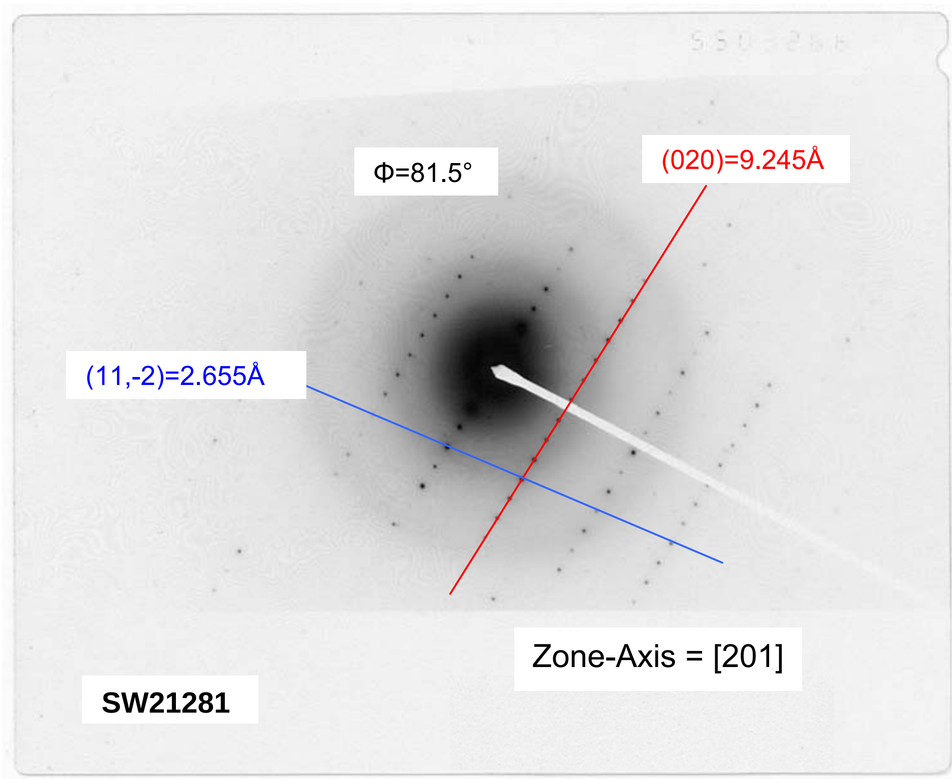

Figure 5. [201] zone-axis SAED pattern of SW21281. We identified this amphibole to be a Libby-type amphibole based on the compositional profile by EDX. The typical "5.2Å to 5.4Å layer lines" are absent at this orientation. However, the "2.6Å" layer lines are striking visible. Even if we do not spend much time to look for a zone-axis pattern, the typical "2.6Å layer lines", which are characteristic of asbestosform amphiboles, can be relatively easy observed without much searching. The objective aperture can be easily calibrated to provide a quick evaluation of the d-spacing of layer lines on the fluorescence screen.

References

Wylie, A.G. and Verkouteren, J.R. (2000) Amphibole asbestos from Libby, Montana: Aspects of nomenclature. American Mineralogist, 85, 1540-1542.