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CRYSTALLINE MASSICOT ON SHEET LEAD, WASTE DUMP, GETCHELL MINE, NEVADA

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Abstract

Crystals of massicot have been found growing on the surface of sheet lead in the waste dump at the Getchell mine, Nevada. The unit cell, refined on 16 well-resolved powder diffraction lines, is orthorhombic *Pbma*, with $a = 5.497(7)$, $b = 4.761(11)$, $c = 5.919(8)$. Spectrographic analysis of several crystals shows them to contain traces of copper and chromium, as does the underlying lead. The measured density is 9.53(2) and the Vickers micro-indentation hardness is 27(6). The dominant crystal form is platy {001} with extended parallel growth on {100} which, together with an unequal development of {110}, gives the larger crystal masses a somewhat ragged appearance. The only other forms noted are the very rare development of {010} and {111}. The crystals frequently contain randomly distributed spherules of secondary lead and, more rarely, thin tabular rods of lead which are oriented parallel to the [[110]] direction of the massicot.

Occurrence and Origin

Crystals of the yellow orthorhombic polymorph of lead oxide have previously been known only in artificial systems, the habit of all natural material being massive to scaly incrustations, usually occurring on galena. The specimens described here therefore represent the first reported occurrence of massicot in well-defined natural crystals.

The two specimens of massicot examined, which were sent to the author for identification by the Mineralogical Research Company of San Jose, California, are said to have been collected in 1959 at the Getchell mine, Humboldt Co., Nevada. The provenance of the material is, however, open to some question, and there is considerable doubt that it represents a strictly natural occurrence.

The Getchell mine, known to mineralogists primarily for its sulphides of antimony, arsenic and mercury, is basically a gold-mining operation. The gold occurs as submicroscopic grains in a fault gouge composed of minute quartz crystals and amorphous carbon (Joralemon 1951). At least eight sulphide minerals from this mine have been documented (Joralemon 1951; Weissberg 1965), but no record exists of any lead-bearing species from the property. The collector of the massicot specimens reported that they were found in the talus of what is probably the south pit extension area. If, as is not unlikely, there was a fire assay laboratory on the Getchell property during some early stage of the mining operation, many of the peculiarities of these specimens can be explained.

The larger of the two specimens, some 2 × 2 × 5 cm, weighs just under 100 grams. It consists of crumpled and compacted sheets of lead, which are coated with a thin film of an orange powder and enclose large flat crystals and crystal cleavages of a yellow mineral similar to orpiment. When the specimen was partly unfolded, several small lumps of glassy black scoriaceous material were found. Spectrographic analysis of the sheets of lead showed them to be essentially pure, entirely free of silver but with trace amounts of copper (0.1%) and chromium (0.2%). The diffraction pattern for a fragment of this material matched perfectly the pattern for lead in the Powder Diffraction File of the Joint Committee on Powder Diffraction Standards and could be fully indexed for $a = 4.96$Å. The scoriaceous material proved to be an *x*-ray amorphous glass; a spectrographic analysis showed major Ca, Al, Si, Fe and Pb. A minute bleb of crystalline white material found enclosed within one of the scoriaceous lumps gave a calcite powder pattern.

The second specimen, about 2 × 1 × 1 cm and weighing 13 grams, yielded similar results on analysis.

It is probable that these specimens resulted from the weathering of discarded assay lead, intermingled with small lumps of an acid slag; their natural presence in the mine area would be most unlikely.

Description of the Massicot

The massicot occurs in two forms on these specimens. There is a thin layer of submicroscopic orange powder coating all exposed portions of the lead sheets and the exteriors of the lumps of slag. Most of the massicot is, however, in the form of well-developed orthorhombic tablets. The crystals have grown parallel to the local surface of the lead or between layers of lead in the interior of the folded mass. They are loosely attached to the underlying lead and are frequently curved or crinkled in conformation with it. Well-terminated crystals of massicot rarely extend as free-standing individuals into hollows between the folds (Fig. 1).

The crystals are invariably flattened parallel to {001} and frequently form extensive platy aggregates of crystals in parallel growth. The ragged appearance of the edges of many of these crystal aggregates is a result of unequal development of the form {110} in adjacent member crystals. There is a well-developed {001} cleavage in the plane of the plates. The crystals are sectile and quite flexible, but their inelasticity causes them to develop a crackle pattern along any zone of flexure (Fig. 1). They have a pearly to greasy lustre of the cleavage surfaces, but appear uniformly dull and somewhat powdery on undisturbed crystal faces. The mineral is orpiment yellow-orange with occasional traces of a reddish tint on the {*hk*0} faces, and is translucent in thin crystals. Cleavage flakes are a transparent sulphur yellow.

The Mohs hardness is about 1.5. Several attempts were made to measure the Vickers micro-indentation hardness (1 gram load) on thick crystals. The previously mentioned crackle effect rendered the values obtained somewhat uncertain ; however, a range of 18 to 46 with a mean of 27 was found for 15 determinations. The powder pattern of the massicot is given in Table 1, together with unit cell data obtained from Weissenberg photographs and refined on the basis of 16 well-resolved powder diffraction lines. The values compare favourably with those for artificial PbO (PDF card 5-570). Seven crystals weighing about 20 mg each were carefully examined under transmitted light to ensure that they were completely free of secondary lead inclusions, and were subsequently used for specific gravity determinations. The average specific gravity obtained for the seven crystals was 9.53, with a range of 9.45 to 9.57 ; this agreed well

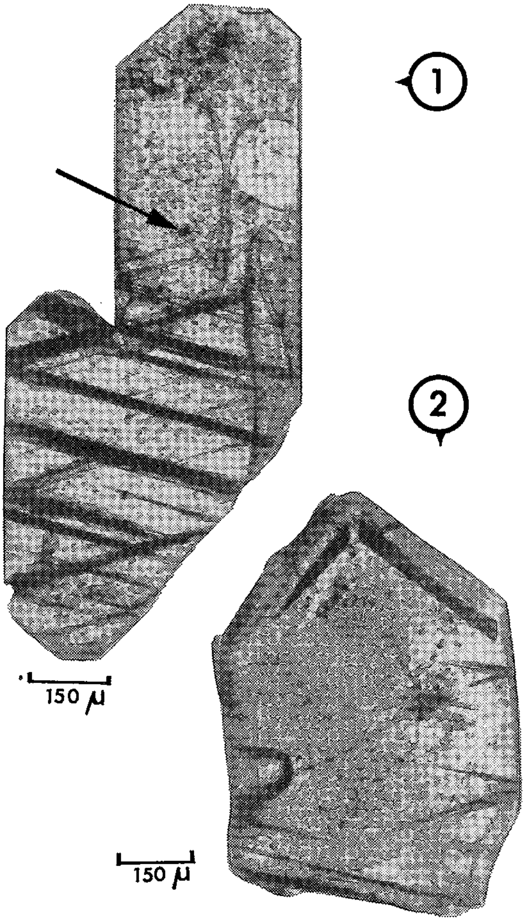

Fig. 1. View perpendicular to (001) of a composite crystal of massicot, taken from a pocket in the interior of the larger specimen. The (110) face (upper left of the larger crystal) is clearly longer than the (1$\bar{1}$0) face (upper right). The development of the horizontal (010) face between these two is extremely rare, all but two of the crystals examined terminating without a pinacoid. The diagonal dark bands across the lower half of the crystal are the expression of the crackle pattern caused by flexure producing internal motion parallel to the excellent (001) cleavage. The speckled appearance of the crystal is in general due to a dusting of powdery massicot over its surface ; however, a few small lead spherules may be seen within the crystal (arrow).

Fig. 2. View of a single crystal of massicot showing the more common termination. The black bands parallel to the [[110]] direction are tabular rod-like inclusions of lead which have been overgrown by later-stage massicot.

TABLE 1. POWDER DIFFRACTION AND CRYSTALLOGRAPHIC DATA FOR MASSICOT FROM THE GETCHELL MINE, NEVADA

I	d_{meas}	d_{calc}	hkl
1	5.895	5.919	001
10	3.075	3.075	111
2	2.950	2.960	002
4	2.752	2.749	200
3	2.495	2.493	201
1	2.366	2.380	020
1/2	2.208	2.209	211
1	2.010	2.014	202
1/2	1.969	1.973	003
5	1.860	1.857	103
1	1.793	1.800	220
2	1.719	1.721	221
3	1.643	1.643	311
3	1.540	1.538	222
1	1.471	1.476	131
1	1.402	1.410	321

a=5.497(7), b=4.761(11), c=5.919(8); space group *Pbma* with the value of 9.57, calculated on the basis of 4[PbO] in the unit cell.

DISCUSSION

Minute spherules of lead are commonly scattered at random throughout a massicot crystal (Fig. 1). These lead spherules rarely reach 0.5 mm in diameter, and then protrude from the (001) faces of the crystals. Spherules up to 1 mm in diameter and flattened on two parallel sides are occasionally found attached to the (001) faces of large massicot crystals.

An interesting feature of several of the massicot crystals is the presence of oriented inclusions of lead (Fig. 2). These inclusions are in the form of narrow tabular rods ($\simeq 300 \times 25 \times 5$ μ) lying parallel to the [[110]] direction of the massicot crystals. They are always completely enclosed by massicot, and are frequently found some distance into the crystal, probably indicating the overgrowth of massicot over secondary lead. Calculations based on the structure of artificial PbO (Kay 1961) suggested that massicot and lead could share a common (220) plane without major displacement. A tiny fragment of the rod-like lead inclusions, mounted to rotate about the postulated [110] axis, gave a badly smeared rotation photograph, as was to be expected from the distortions caused in cutting out the fragment. However, a measurable period of 1.76Å would appear to confirm the hypothesis that the two minerals share a common orientation. All these secondary forms of lead either are contemporaneous with the massicot or formed after large massicot crystals had grown.

Garrels & Christ (1965, Fig. 7.26b) present an Eh-pH diagram of the stability relations among lead compounds in water, assumed to contain $10^{-1.5}$ (molal) total dissolved carbonate ion, at 25°C and one atmosphere total pressure. In the diagram there is a very narrow vertical stability field for massicot at pH = 13, lying between Eh +0.2 and −0.5. If the Eh exceeds +0.2, minium (Pb_3O_4) will form at the expense of massicot; if the Eh falls below −0.5, lead will form. If the pH falls below 13, between the Eh limits given, hydrocerussite, $Pb_3(CO_3)_2(OH)_2$, will form. The two specimens were therefore very carefully examined for traces of minium and hydrocerussite, as it had been determined that neither galena nor cerussite was present. On one end of the larger specimen, adjacent to a slag inclusion, there was a small area of brilliant scarlet crystals. Under high magnification it was seen that these crystals were massicot which had been surficially altered to a red mineral. A powder mount of a minute quantity of the red material gave the pattern of minium. The presence of minium may account for the reddish tint observed on {110} of some massicot crystals. On the surface of the adjacent slag inclusion, six tiny greenish hexagonal(?) tablets were developed in addition to the ubiquitous powdery massicot. These tablets were also *x*-rayed, giving the powder pattern of hydrocerussite as well as traces of the three strongest lines of the massicot pattern ; this suggests that the hydrocerussite formed by alteration of previously existing massicot.

The orebody at the Getchell Mine lies in a fault zone cutting a limestone-argillite sequence, and is itself cut by late stage calcite veins (Joralemon 1951). Within such an environment the necessary Eh-pH conditions would have been present, given probable changes in the water table due to the mining operation, for all four of the minerals found to be stable at various times. Since only very minor changes in Eh are necessary to render the lead unstable with respect to the massicot, the presence of several stages of secondary lead overgrown by massicot is not unexpected. The bulk of the lead would, of course, have been protected by the layer of previously formed massicot.

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