



## Characterization of Gold Crystallinity by Diffraction Methods

The term *crystal* is used in two related but distinct contexts in mineralogy. First, it is used to describe a mineral or other solid whose natural shape comprises flat planes that are arranged in a symmetric order. In other words, it is used to describe the morphology of a solid. The term *crystal* is also used to denote a regular and repeating arrangement of atoms in three dimensions. The macroscopic formation and geometry of a crystal are in large part the product of the microscopic atomic structure. Ultimately, it is the regular, repeating arrangement of atoms that makes a solid material a crystal.

**Figure 1.** Gold crystals from Icabarú, Bolívar, Venezuela. The crystal at the bottom right measures  $1.4 \times 1.1 \times 1$  cm and weighs 5.3 grams.

Morphologically, crystals can be euhedral, composed of flat faces on all sides; subhedral, not completely bounded by crystal faces; or anhedral, exhibiting no crystal faces at all. The adherence to an ordered, repeating arrangement of atoms within a crystal can be quite variable; as a result, a continuum from almost perfect to very poor order (*coherency*) can be found in natural crystals. The extent to which atoms are ordered is known as the degree of crystallinity. Some

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**Figure 2.** Gold crystal from Icabarú, Bolívar, Venezuela. It is also shown in the bottom right of figure 1. The front, hopped face is the one that produced the diffraction pattern in figure 8b.

minerals, such as fluorapatite, are most commonly found with an extremely high degree of crystallinity. Other minerals, such as smectite, are almost always poorly crystalline (have a low degree of crystallinity). Graphite exhibits the complete range of crystallinity, from very well ordered to essentially amorphous (no atomic order). There is an observable trend between the degree of crystallinity and the hardness of minerals, as observed by Paul Moore (pers. comm. to John Hughes). Generally, soft materials have a greater tendency to be poorly crystalline. These two properties, of course, are related to the nature and strength of bonds between atoms within a crystal. It is, therefore, not surprising that such a correlation exists.

Gold is a soft mineral and is commonly found as anhedral crystal masses. Specimens with euhedral crystals are less common. Natural gold crystals, as single or polycrystalline aggregates (composed of many crystals), are found in situ in quartz veins and other types of primary deposits (Cook et al. 2003; Huizing 2004; Wilson 1987; and references therein). Single gold crystals exhibiting recognizable crystal forms are not common and rarely exceed 2 cm in size. Gold belongs to the hexoctahedral crystal class,  $\frac{4}{m}\frac{2}{m}\frac{2}{m}$ , and most forms within this

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**Figure 3.** Gold crystals from Icabarú, Bolívar, Venezuela. Crystal no. 1 measures  $2.4 \times 2.2 \times 1.0$  cm and weighs 17.8 grams. Crystal no. 4 measures  $2.16 \times 2.11 \times 2.06$  cm and weighs 73.2 grams.

class have been observed in gold (Francis 2004; Weiner 2003). Gold crystals are also frequently hopped and distorted.

Gold is also commonly found in placer deposits. Herein, the gold has been weathered from its original host rock, transported by streams or rivers, and deposited with sediment where flow velocities are no longer sufficient for transport (Webber, Hochleitner, and Cook 2003; Rakovan 2004). Because of the malleability of gold and the mechanical forces involved in bed-load transport in rivers, placer gold is usually highly deformed and found in nugget form. Gold from some placer deposits, however, may show distinct crystal morphologies, indicating smaller degrees of physical deformation during weathering and transport. Low levels of physical deformation indicate a short travel distance. In contrast, some have speculated that gold can form in situ in placer sediments. There is, however, great controversy over the ability of gold to form large masses in this way. In the few published studies of the internal structure of large gold masses, the samples investigated were shown to be alluvial and to have formed at high temperatures (Hough et al. 2007 and references therein). If large gold crystals can form directly in placers, it is likely that they will have undergone little mechanical disturbance. Other types of gold deposits do exist, and a complete coverage of all types can be found in Webber, Hochleitner, and Cook (2003).

Because of the rarity of large well-formed crystals of gold, their values can be quite high on the collector market. This has led some to create forgeries, often quite sophisticated, of large, single gold crystals. It has been a challenge to find definitive ways to verify the authenticity of gold crystals by nondestructive means, and little is published on the subject. We have used X-ray and neutron diffraction to study morphologically sharp, well-formed samples of gold from several reported deposits. These samples have morphologies that suggest that





**Figure 4.** Gold crystals from Russia. The upper crystal measures roughly 1 cm across.

they grew as single crystals. X-ray diffraction data from these samples, however, are surprising and indicate polycrystallinity, which is inconsistent with their morphology. Such data may be interpreted by some to indicate a forgery. However, it is shown below that this is not the only way to explain the inconsistency and that methods other than X-ray diffraction are necessary to test the authenticity of the crystals.

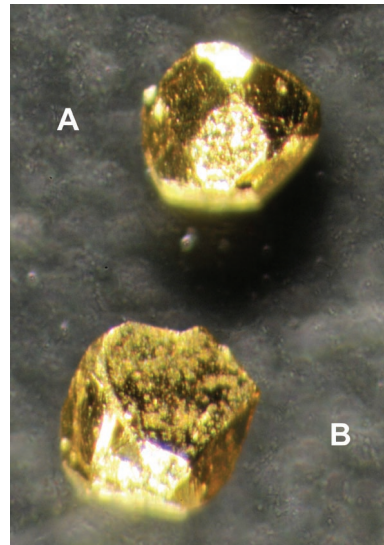
### Analysis of Gold Crystals

#### Samples

Samples from three different reported locations have been investigated in this study. The first and largest suite is from Icabarú, Bolívar, Venezuela (Wilson 1987b). These crystals were all collected from placer deposits. Twenty individual crystals from Icabarú, most pictured in figures 1, 2, and 3 were also examined by X-ray diffraction. Four of these crystals (nos. 1–3 in fig. 3 and the crystal in fig. 2) were examined by neutron diffraction. The crystals range in size from 0.75 cm to about 2.5 cm in maximum dimension. Most are hopped single crystals with the octahedral form  $\{111\}$  dominant, but faces on the two largest crystals are not hopped and are of non-octahedral forms (e.g.,  $\{311\}$ ).

Two individual samples (fig. 4) from an unknown location and deposit type reportedly in Russia were also examined by X-ray and neutron diffraction. The crystals are equant with dimensions of approximately 1 and 1.5 cm. Their morphology is unusual and resembles iron-cross twins found in pyrite (Hahn and Klapper 2006). Small quartz crystals are attached to both gold crystals.

Finally, twenty small (0.5–2-mm) single crystals from Bear Mountain, near Harrison Hot Springs, British Columbia, Canada, were examined by X-ray diffraction. These crystals



**Figure 5.** Gold crystals from Bear Mountain, near Harrison Hot Springs, British Columbia, Canada. The upper crystal measures 1.5 mm across.

were collected from the deposit in which they formed and have not been mechanically disturbed by stream transport. The occurrence consists of hydrothermal quartz veins hosted by a coarse-grained

igneous rock. All of the gold crystals were found in cavities within the quartz veins. These cavities were filled by calcite after gold crystallization. To remove the gold, dilute HCl was used to dissolve the calcite. Most of the gold crystals were not attached to quartz in the cavities and freely fell out once the calcite was removed. The crystals are morphologically sharp with varying degrees of luster (fig. 5).

#### Diffraction

*Diffraction* is an interference phenomenon that occurs when a wave encounters an obstacle to its propagation. When a group of such obstacles is periodic in its arrangement, the resultant diffraction is a complex wave pattern that contains information about the arrangement of the obstacles. Diffraction can occur with all waves, including water, seismic, sound, and electromagnetic waves. The effects of diffraction are generally most pronounced for waves with wavelengths that are similar in dimension to the size and spacing between diffracting objects.

All electromagnetic radiation can be considered as waves. The difference between photons in different parts of the electromagnetic spectrum—e.g., radio waves, visible light, and X-rays—is that they have different wavelengths (and energies). It turns out that X-rays have a range of wavelengths that in part is similar to the size of atoms and the distances between them in crystals. Thus, X-rays can be used, through the process of diffraction, to study the atomic arrangement of crystals and their degree of crystallinity. Subatomic particles such as electrons, neutrons, and protons also have wavelike properties, and their wavelengths can be similar in size to atoms and their separation in crystals, depending upon the energies of such particles. Consequently, these particles may also be diffracted from crystals to yield information about their crystallinity.

Although the physics of diffraction is the same for all waves, the characteristics of a diffraction pattern depend on the geometry of the instrument used to collect it and the nature of the sample. Three different diffractometers were used in this study, and the data from each look slightly different. Furthermore, for any given instrument there are distinct

differences between the diffraction pattern that is created by a single crystal and that created by a polycrystalline sample.

### X-ray Diffraction

All of the Venezuelan and Russian samples, because of their size, were X-rayed on a Scintag PadX diffractometer with Bragg-Brentano geometry using Cu  $K\alpha$  radiation at Miami University in Oxford, Ohio. This instrument is ideally designed for the diffraction analysis of flat polycrystalline (e.g., powder) samples. It can, however, be used to X-ray single crystals. The size range of these samples (0.75–2.5 cm) is ideal for mounting on the instrument's sample-mounting stage (not too large) and for producing a strong diffraction pattern from the large area incident X-ray beam (not too small). Crystals were mounted in a  $2.6 \times 2.6 \times 1.5$ -cm leucite box with an open top and one open side. A crystal was set into a wad of molding clay so that it protruded above the open top of the box. A flat plate was used to push the crystal into the clay so that one crystal face was perfectly parallel with the open top and exactly aligned (in height) with the edge of the box along the open top (fig. 6). When the box was set into the sample-mounting stage of the diffractometer, the oriented crystal face was in the proper orientation for diffraction and was closely aligned with the diffraction circle of the goniometer.

Diffraction data from single crystals oriented in this way on a diffractometer with Bragg-Brentano geometry would ideally exhibit very intense peaks corresponding to diffraction from the oriented crystal plane and the absence of diffraction from other planes. In the normal case of a polycrystalline sample, one with a random orientation of many crystals (e.g., a powder), the result would be a diffractogram exhibiting diffraction peaks from many crystallographic planes. Crystallographers use Miller indices  $\{hkl\}$  to identify different crystallographic planes. This was tested on single crystals of several other minerals, including calcite  $\{104\}$ , garnet  $\{110\}$ , and pyrite  $\{100\}$ , and was confirmed to be true. Figure 7 shows the X-ray diffractogram collected from a single crystal of calcite with a  $\{104\}$  cleavage surface oriented in the horizontal and aligned with the diffraction circle of the goniometer. The large peak at approximately  $29.5^\circ$  ( $2\theta$ ) is the result of X-ray diffraction from the  $\{104\}$  set of atomic planes in the crystal. If the diffractogram was collected to larger angles, the presence of higher-order diffractions from this set of planes (i.e., 208) would be expected. The narrow lines throughout the diffractogram indicate the ideal position of all diffractions expected for a powder sample. These are all absent in this single-crystal diffraction pattern.

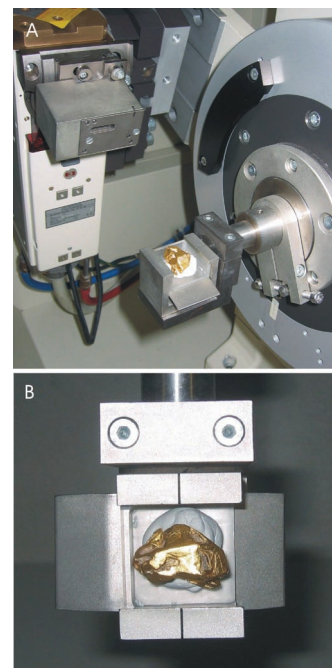
Most of the gold crystals exhibit an octahedral form; consequently,  $\{111\}$  faces were oriented in the diffraction plane for data collection. For each crystal at least one face was oriented, and diffraction data were collected. For many of the crystals, however, diffraction data were collected for numerous oriented faces. In all but one gold crystal examined from Venezuela, the diffraction patterns include all peaks expected for a polycrystalline sample. In many cases the relative intensities of the diffraction peaks are very close to that expected for a polycrystalline material with random orienta-

tion of crystals (fig. 8a). For the well-formed trapezohedron  $\{311\}$  with twenty-four faces (no. 4 in fig. 3), ten of the faces were X-rayed; surprisingly, all yielded polycrystalline patterns. This seems contradictory to the single-crystal morphology of these samples, bringing into question their authenticity. However, several X-ray diffraction experiments (see below) indicate that the samples may truly be natural single crystals, even though their X-ray diffraction patterns indicate that they are polycrystalline.

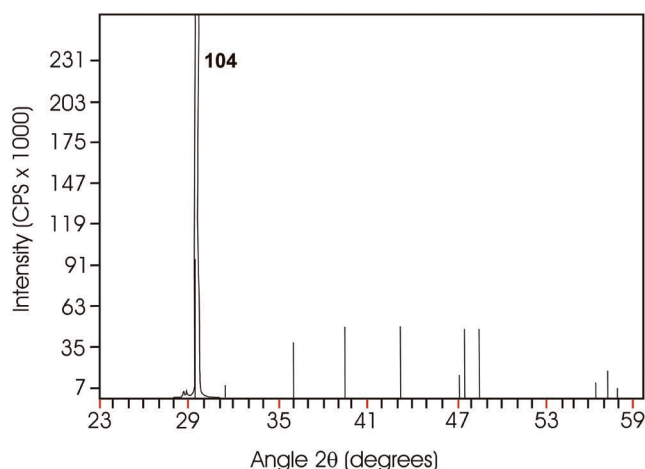
A key sample from Venezuela yields polycrystalline diffraction patterns from all but one of its octahedral faces. One deeply hopped face, however, yields a pattern with strong 111 and 222 diffraction peaks and very weak diffraction peaks from non- $\{hhh\}$  crystal planes (fig. 8b). This pattern indicates that most of the diffracting volume is structurally coherent (i.e., a single crystal), but a small portion is composed of crystals or crystal domains with different orientation. From this observation we hypothesize that the polycrystalline X-ray diffraction pattern produced by most placer gold crystal faces is the result of mechanical deformation (*cold working*) of the surface, which transforms the surface of the crystals into a polycrystalline mosaic. Such cold working—if it does exist—is likely the result of pounding and abrasion associated with stream transport. We speculate that the lack of structural deformation (and polycrystallinity) on the one, deeply hopped face of the unique crystal is the result of fewer mechanical interactions during transport. Likely, the morphology protected this face, preserving the internal single crystallinity.

### Tumbling Experiments

To confirm that gold samples exhibiting single-crystal diffraction patterns do exist, we looked for morphologic single crystals that were extracted from their original environment of formation and that had not been mechanically deformed. The samples from British Columbia are small, very sharp, single crystals that formed in open spaces (pockets) by precipitation from solution (fig. 5). They were chemically extracted from their encasing calcite with great care so as not to mechanically disturb them



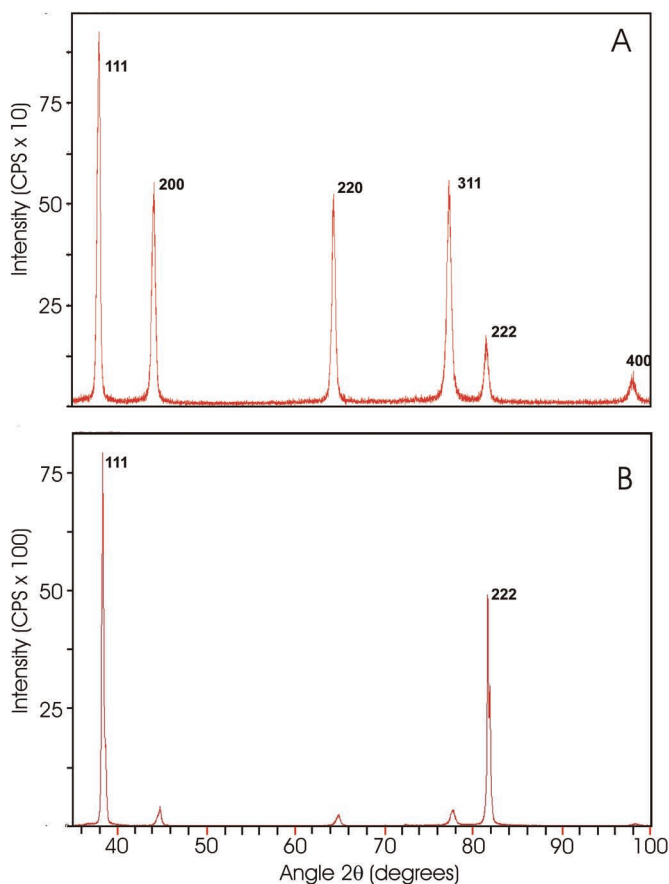
**Figure 6.** Sample mounting box with oriented crystal on the Scintag PadX diffractometer sample-mounting stage. (a): Oblique view showing gold crystal and X-ray source behind. (b): View directly down onto one crystal face and top of sample-mounting stage. Notice reflections from the two, indicating that they are parallel.



**Figure 7.** X-ray diffractogram (Scintage instrument), a plot of diffraction intensity as a function of angle (measured in  $2\theta$ ) collected from a single crystal of calcite with a (104) cleavage surface oriented in the horizontal and aligned with the diffraction circle of the goniometer. The large peak at approximately  $29.5^\circ$  ( $2\theta$ ) is the result of X-ray diffraction from the (104) set of atomic planes in the crystal. Narrow lines throughout indicate the ideal position of all diffractions expected for a powder sample. The slight offset of the (104) diffraction peak from its expected position is due to a small error in the height of the crystal from its ideal position in the instrument.

during handling. These crystals are too small to collect data using the Scintag powder diffractometer but are sufficiently small to analyze with a diffractometer designed to X-ray single crystals. X-ray diffraction data from these crystals were collected on a Bruker® APEX diffractometer with Mo  $K\alpha$  radiation and a CCD area detector (essentially a digital camera). Crystals were mounted on the end of a glass fiber and rotated in the incident X-ray beam while diffracted beams were being collected on the area detector (fig. 9a). Rotation diffraction images show intense, well-defined spots, indicative of single crystals, with the superposition of much weaker partial diffraction rings, indicative of some degree of poor crystallinity. Thus, these morphologic single crystals—which to the best of our knowledge have not been mechanically deformed, and which are known to be natural—show the diffraction characteristics of single crystals with a distinct degree of disorder. This may not be surprising given the hardness of gold.

To test our hypothesis that mechanical deformation from stream transport deforms the surfaces of placer gold, resulting in a polycrystalline mosaic, we tumbled these small, naturally undeformed crystals to mimic the forces associated with stream transport. This was done by rolling them around a brass cylinder with several grains of quartz. Quartz grains were chosen with sizes similar to that of the gold crystal. Tumbling was facilitated by blowing air through an oriented opening in the wall of the brass cylinder. The air caused the crystals to roll around the inner circumference of the cylinder. Crystals were tumbled for several hours at a time, then removed and X-rayed. After only twenty-four hours of tumbling, the gold crystals yielded perfect polycrystalline diffraction patterns (fig. 9b). This test supports the hypothesis that mechanical deformation associated with



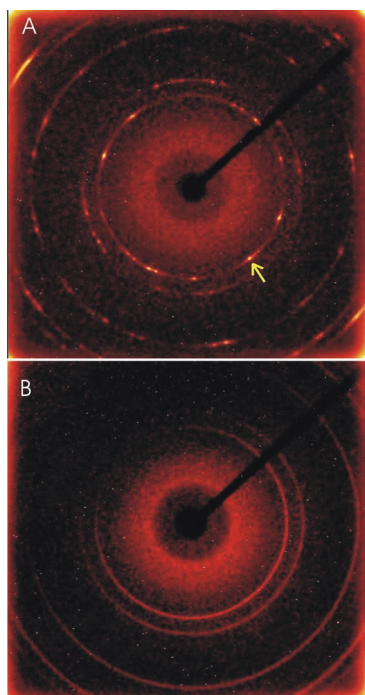
**Figure 8.** X-ray diffractograms (Scintage instrument) collected from a single crystal of gold from Venezuela (fig. 2) with different crystal faces oriented in the horizontal and aligned with the diffraction circle of the goniometer.

stream transport can cause the formation of a polycrystalline surface layer on gold.

Gold is highly absorbing of the X-ray energies used in the diffraction experiments (Cu  $K\alpha = 8.05$  keV, Mo  $K\alpha = 17.48$  keV); consequently, these X-rays do not penetrate very deeply into gold crystals. Therefore, the diffraction signal in the X-ray measurements comes only from the surface of the crystals when they are the size of those used in this study. This leads to a problem in the use of X-ray diffraction to analyze the atomic arrangement of large gold crystals. If a polycrystalline diffraction pattern is obtained, it could be the result of two possible scenarios. First, the surface of the sample may have become polycrystalline by natural cold working. The same diffraction pattern, however, would result if the entire sample were actually a polycrystalline aggregate, as would be the case if the sample had been cast from molten metal.

Although our measurements do not indicate the depth to which cold working will disturb the crystallinity of a gold sample, we hypothesize that the majority of the interior would not be affected in centimeter-sized samples. If this is correct, diffraction from the bulk of the sample, rather than the surface, would yield more conclusive information about the nature of the sample (i.e., polycrystalline versus single crystal). Cutting a sample to expose its interior for X-ray diffraction is problematic for several reasons. It is destructive, and given





**Figure 9.** X-ray diffraction data collected from a single crystal of gold from British Columbia in the Bruker® APEX. (a): Before tumbling. Arrow points to a sharp diffraction spot. (b): After tumbling for twenty-four hours. Very small white spots are defective pixels in the CCD detector.

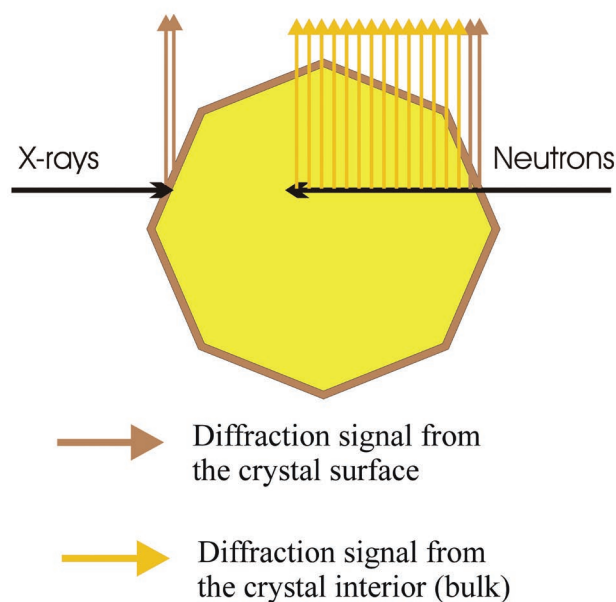
the malleability of gold, the cutting process may itself cause cold working. To avoid both of these problems, neutron diffraction was used to analyze the interior of the crystals.

### Neutron Diffraction

Neutrons of sufficient thermal energy will penetrate and diffract from much deeper levels of the crystals than will X-rays (fig. 10). Neutron diffraction measurements were made on the single-crystal diffraction (SCD) instrument (fig. 11) at the Los Alamos Neutron Science Center (LANSCE), Los Alamos National Laboratory (LANL), New Mexico. Even though gold has a relatively high absorption cross section for neutrons, calculations indicate that neutrons can penetrate 1 cm or more into gold. Hence, the center of even the largest crystal studied can be reached. Just as with the X-rays, the nature of the neutron diffraction patterns gives us information about the crystallinity of the samples. Because of the penetrating power of the neutrons, however, the diffraction patterns are the result of a much greater volume of the crystal, most of which lies well beneath the surface, where natural cold working is hypothesized to be confined.

Six crystals were analyzed by neutron diffraction. These are the hopped octahedral crystal that exhibited a single-crystal X-ray diffraction pattern on one of its faces (fig. 2); three of the large crystals (nos. 1–3) pictured in figure 3; and the two Russian crystals (fig. 4). The large trapezohedral crystal from Venezuela (no. 4 in fig. 3) was not analyzed by neutron diffraction. All of the Venezuelan samples analyzed gave diffraction patterns with well-constrained spots similar to that shown in figure 12. This is clear indication that the samples are true single crystals. No obvious indication of streaking in the SCD pattern is seen (elongation of diffraction spots, approaching a polycrystalline ring pattern, fig. 12), which suggests that the vast majority of the diffraction volume was coherent and that the volume of the cold worked surface is very small (undetectable by this experiment).

In contrast to these diffraction patterns, those collected for the two purported Russian samples exhibited diffraction rings. This indicates that the samples are polycrystalline

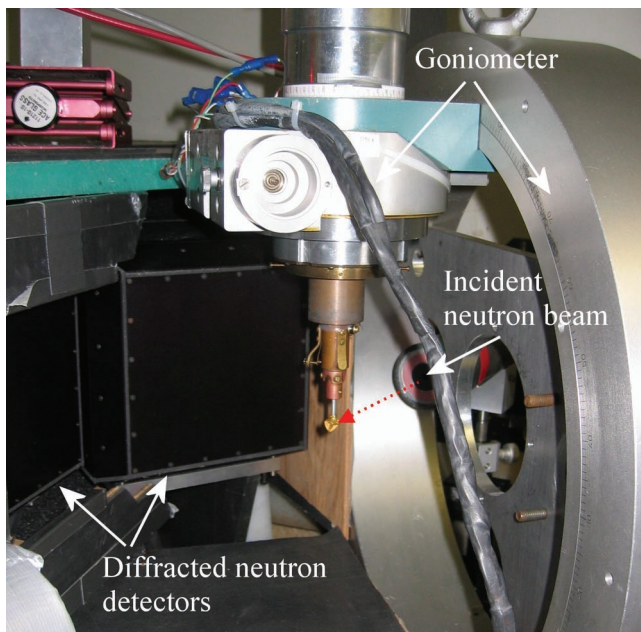


**Figure 10.** Schematic showing the relative difference between X-ray and neutron penetration into gold crystals and the resulting portion of the crystal involved in diffraction.

throughout, with no indication of large single-crystal domains. Because of the poor signal to noise in data collected from the polycrystalline Russian samples on the SCD instrument, a second dataset was collected on the high-pressure-preferred orientation (HIPPO) instrument at LANSCE. This was designed specifically for diffraction analyses of polycrystalline samples, so although we did not utilize the high-pressure capabilities, it was better suited for these samples. An example of the HIPPO neutron diffraction data from Russian sample no. 1 is shown in figure 13.

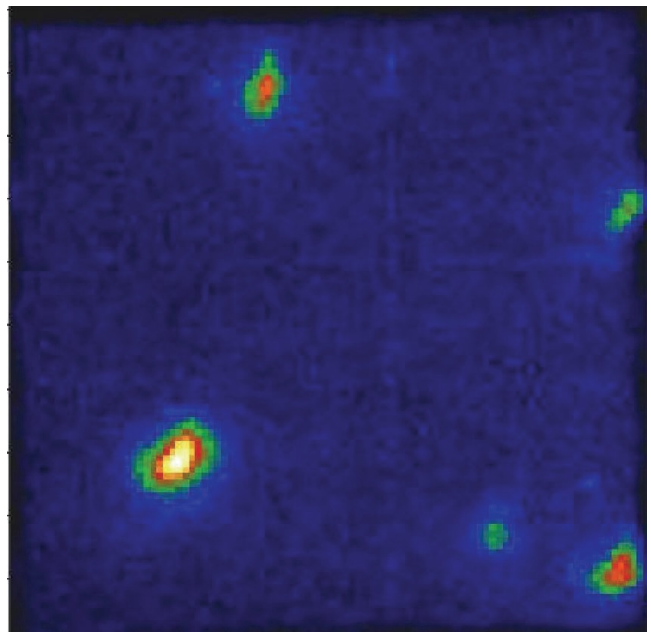
### Discussion

Because diffraction of a wave is dependent upon the spacing and arrangement of obstacles with which it interacts, diffraction data can be used to study different aspects of crystal structures and the nature of crystalline samples. X-ray diffraction is most commonly used as a “fingerprinting” method for phase identification. It can also be used to determine the arrangement of atoms in a crystal (structure solution and refinement). In this study, X-ray diffraction was used to assess the crystallinity of large (1–3-cm) gold samples that had been mechanically disturbed during natural weathering and transport, as well as naturally undisturbed gold crystals that were tumbled to mimic the deformation associated with river transport. A limitation in the use of X-rays to study large samples of gold is that the depth of penetration is small, rendering the technique suitable for studying the surface of the samples but not the bulk. Neutrons were used as a complement to X-rays to collect diffraction data from the interior of the crystals. The data from these two techniques are simple but powerful in addressing the question at hand: Do the gold samples, which appear to be single crystals based on morphology, exhibit diffraction consistent with

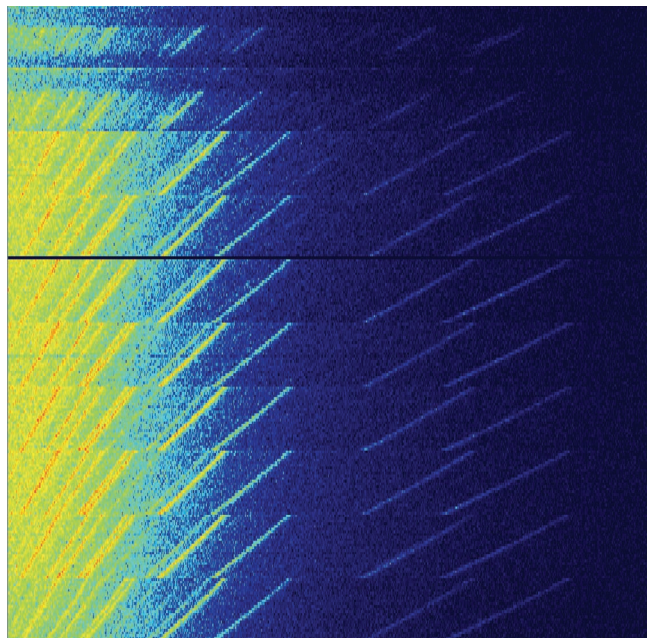


**Figure 11 (above).** The experimental setup on the single-crystal diffraction (SCD) instrument at LANSCE, Los Alamos National Laboratory.

**Figure 12 (upper right).** Neutron diffraction data collected on the SCD instrument at LANSCE, Los Alamos National Laboratory, from the Venezuelan sample pictured in figure 2. Well-constrained diffraction spots indicate that the sample is a single crystal.



**Figure 13 (lower right).** Neutron diffraction data collected on the high-pressure-preferred orientation (HIPPO) instrument at LANSCE, Los Alamos National Laboratory, from the Russian sample (no. 1) pictured in figure 4. The diffraction streaks indicate that the sample is polycrystalline with random orientation of the diffracting domains.



single crystals? The answer to the question can be gotten by simple inspection of the diffraction pattern.

There is a continuum in the nature of diffraction patterns taken from a single-crystal sample with a high degree of coherency, to a sample with a very large number of crystals (a polycrystalline sample) of random orientation. To generalize, a coherent single crystal will give sharp spots on an area detector such as those used in the APEX X-ray diffractometer and the SCD and HIPPO neutron diffraction instruments. At the other end of the spectrum, the sample with a very large number of crystals will yield rings or curved lines (partial rings) of diffraction with equal intensity across a given ring.

The results of X-ray diffraction from the large gold samples from Venezuela and Russia indicate that the surfaces are polycrystalline, which could be the result of mechanical deformation of the samples. This possibility was confirmed by X-ray diffraction patterns collected from small gold crystals before and after deformation by tumbling. In most of the diffraction patterns from the naturally stream-tumbled samples and in the laboratory-tumbled samples, relative peak intensities were consistent with a randomly oriented

polycrystalline sample. This was at first a surprising result because of the common preferred orientation of crystals in cold worked metals. However, given the random nature of applied stresses associated with tumbling in the presence of other particles, it makes sense. All but a single face on one crystal exhibited polycrystallinity; therefore, neutron diffraction data were the key to answering the question of single-crystal authenticity. The four Venezuelan crystals that were analyzed by neutron diffraction are indeed single crystals; the Russian crystals, however, are not.

What do these results tell us about the natural occurrence of the samples? In the case of the large crystals where each has been shown to be atomically coherent throughout its bulk, and therefore a single crystal, we believe that they are natural. This is predicated on the assumption that single

gold crystals (structurally coherent on the atomic scale) of this size have never been synthesized, which is true to the best of our knowledge. As for the Russian crystals, there are several features that indicate that they are not and never were single crystals. The neutron diffraction data are definitive in showing that they are not currently single crystals. It is very unlikely that they were once single crystals and subsequently were mechanically deformed to a polycrystalline state throughout. The sharp morphology of the crystals precludes this possibility. Also, the morphology of these crystals, if that of “iron-cross twins,” is not possible in the  $\frac{4}{m}\frac{2}{m}\frac{2}{m}$  point group to which gold belongs. All of this evidence suggests that these crystals formed by some casting process, be it natural or manmade.

Future researchers using X-ray diffraction to study gold crystals need to be aware that production of a polycrystalline diffraction pattern does not necessarily mean that the entire sample is polycrystalline, and other methods of analysis may be necessary to evaluate the crystalline nature of the sample.

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