CASE STUDY

Infrared Microscopy and Spectroscopy



IR Microscopy Used in Effort to Solve Mystery: Did Mallory Climb Everest in 1924?

By the mid 1920s, the North and South Poles had been reached and the sources of the great rivers had all been discovered. The greatest challenge then remaining was to reach the summit of the world's highest mountain, 29,035-foot Mount Everest. At this point in time, the highest altitude reached by climbers was 24,600 feet and there was some debate about whether man was capable of going higher without running out of oxygen. Thirty-eight year-old George Leigh Mallory was celebrated as

one of Britain's greatest rock climbers and had been on two previous Everest expeditions. Joined by Andrew "Sandy" Irvine, he decided to leave his wife and three small children behind for one more all-out assault that would, if successful, make him one of the most famous people on earth. As Mallory said: "To refuse adventure is to run the risk of drying up like a pea in its shell."

Mallory and Irvine were last spotted in the early afternoon of June 8, 1924 by geologist Joel Odell, who was providing support to their expedition. He said he saw two figures approach and climb a 25- to 30-foot vertical rock ledge at about 27,800 feet that is now called the Second Step. Then clouds swirled in and the figures disappeared, never to be seen again. Some have questioned whether Odell actually saw them climb this difficult obstacle, which in all the years since has only been climbed by one other person without the aid of a ladder or rope from above. If he was correct, Mallory and Irvine would have had a relatively easy climb from there to the summit.



Climbing's greatest mystery

To this day, no one knows whether Mallory and Irvine died on the way up or on the way down. Sir Edmund Hillary and Sherpa Tenzing Norgay later became the men credited as the first to climb Everest in 1954 by a different and easier route. The route taken by Mallory was climbed for the first time in 1960 by a Chinese expedition. In 1979, another Chinese climber named Hongbo Wang confided to a companion that five years before he had found a body. The body appeared to be an Englishman who had been lying there many years.

Wang was killed the next day in an avalanche without providing specific directions on where the body could be found. But the people that were with him in 1979 stated that the only time he was out of their sight was when he took a 20-minute walk from the group's campsite. An expedition led by Eric Simonson finally set out in 1999 to the remote point where the Chinese group had camped. After reaching the site, the expedition fanned out in a 10-minute walk radius from the campsite to conduct their search. Their goal was not only to recover the bodies of the two climbers but also the camera they carried which might hold the answer to climbing's greatest mystery.



Figure 1. Front and back of the section of Mallory's jacket that was recovered from Everest. The photos were taken at the Textile Conservation Centre before any detailed analysis or conservation work on the garment was carried out.

Mallory's body found but not camera

The expedition found Mallory's body but did not find Irvine or the camera. It was clear from the condition of the body that Mallory and Irvine had been roped together and had taken a bad fall. The expedition recovered a DNA sample that was later used to verify the body's identification and samples of his clothing and equipment. Then, because it would have been dangerous to attempt to bring the body down, they held a service and buried the body.

Since the camera that might answer the question one way or the other has not yet been found, investigators have focused on the clothing and equipment found with the body. The clothing in particular is interesting because it might help determine whether the climbers were dressed warmly enough to have a chance of reaching the summit. The style and construction of the clothing was generally identifiable, but for many of the garments, the component fibers could not readily be identified.

Experts brought in to examine clothing

Mike Parsons and Mary Rose, on behalf of the Mountain Heritage Trust, made the decision to bring in experts from the Textile Conservation Centre (TCC), Winchester, United Kingdom, one of the world's leading institutions focusing on the care and interpretation of historic textiles. "There have been suggestions that Mallory and Irvine failed to come back because their clothing wasn't up to the job," said Paul Garside, Research Fellow at the TCC. "We wanted to find out exactly what Mallory was wearing so that mountaineering experts could test his gear and make educated guesses about their ability to reach the top. We weren't asked to comment on whether or not we thought it likely that Mallory made it to the top – it's rather out of our field. However, one of the aims of the research was to attempt to determine if his equipment was 'state of the art' for the period, as there had been some suggestions that one of the reasons he might have died on the mountain was that his clothing wasn't up to the task."

Garside began by examining the fabric samples with a light microscope, but could determine very little information by this method because of the fabric's deteriorated state. "In this situation, which is very common with historic textiles, I normally turn to Fourier Transform Infrared (FT-IR) Microscopy which provides a unique chemical fingerprint of any type of fabric material," he said. "Then I can use this fingerprint to match a deteriorated fabric against another one that is in good enough condition to be identified."

Unique capabilities of FT-IR

FT-IR quickly provides a unique fingerprint of a complicated material made up of multiple constituents. Another advantage of this technique is that it requires little or no sample preparation and that it can generate spectra in a matter of a few minutes. An IR microscope provides a valuable add-on that makes it possible to analyze small textile fragments without any sample preparation.



Figure 2. Paul Garside examines textile samples with the PerkinElmer Spectrum One spectrometer fitted with a modified PerkinElmer Universal ATR accessory and to the left, a PerkinElmer AutoIMAGE microscope.

The principle technique used was attenuated total reflectance (ATR) spectroscopy, using the PerkinElmer* Spectrum™ One¹ FT-IR instrument along with a Universal ATR accessory also produced by PerkinElmer. Garside also used the PerkinElmer AutoIMAGE™² microscope for some of the samples, especially those that were particularly small or in a fragile, fragmentary condition. "Its high signal-to-noise ratio makes it possible to generate clear spectra from samples as small as 10 microns, smaller than what most other instruments can handle," Garside said. "The Spectrum One and AutoIMAGE work together as if they were a single instrument, which makes them easier to use. Finally, the PerkinElmer software makes it very easy to create a reference library and automates the process of comparing new spectra to its contents."

Positive identification of the clothing samples

Garside received samples of the jacket, pants, and three different shirts found on Mallory's body. He simply placed the samples on the crystal window of the instrument and applied a slight pressure to hold them against the window. With conventional FT-IR, it would have been necessary to grind the sample into a powder, mix it with a carrier such as mineral oil, and then form it into a mold or a compression disk. "The elimination of sample preparation saves time and means the sample doesn't incur any damage," Garside said.

He matched up the spectra of most of the materials to samples in his library. But the spectra of a few of the samples were close to several materials that have very similar spectra – cotton, linen and hemp. In order to make a positive identification, Garside used the PerkinElmer Infrared Polarizer kit to study the orientation of polymer chains. With a polarizer, infrared spectra are generated only when the chains are oriented with the polarizer. Despite their similar infrared spectra, these materials all have distinct angles of orientation. Garside concluded that the pants and jacket were made of cotton and treated with weatherproofing. The shirts were made of wool and silk. "The clothing worn by Mallory was basically as good as anything available at the time of the climb, although it didn't provide nearly as much protection as the clothing worn by today's climbers," Garside said.

Examining multiple layers

Although it wasn't necessary in this application, Garside has also begun using the PerkinElmer Near Infrared Reflectance Accessory (NIRA) which is available with a probe that makes it possible to obtain distinct spectra from the different layers of multiple layer fabrics. NIR spectroscopy provides penetration into the sample to a depth of several millimeters. The contribution of the inner layer is normally very weak and often invisible in the initial examination. Garside records a second spectrum solely of the outer layer and subtracts it from the combined spectrum, yielding the spectrum of the inner layer. Garside said he has been using NIR spectroscopy recently to look at foam materials that were produced in the 1950s and 1960s and are just beginning to come into museum collections.

"Even though IR microscopy didn't solve the mystery all by itself, it provided information that has been very useful to the researchers working on this problem," Garside concluded. "We weren't asked to comment on whether or not we thought he reached the peak, but rather to determine the composition and construction of the garments, and make comparisons with what was known to be available at the time. On balance, our evidence showed that they were wearing the state of the art in cold weather gear as it existed at the time of their climb. Climbing experts tell us that with this gear, it would have been possible for them to summit. But further physical evidence will have to be found before we can definitively conclude whether they made it or not."

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 $^{1\} Spectrum\ One\ has\ been\ superceded\ by\ the\ PerkinElmer\ Frontier\ FT-IR\ system.$

² AutoIMAGE microscope systems have been superceded by the Spotlight 200 FT-IR microscope systems.