Journal of the Korean Conservation Science for Cultural Properties
Vol. 5, No. 2, 1996
Printed in the Republic of Korea

Scientific Studies on Ancient Silk Fibers Used for Textiles Excavated at Archaeological Sites in Japan, Using Microscopic Fourier Transform Infrared Spectroscopy

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□ ABSTRACT : Silk fibers excavated at Fujinoki mounted tomb, Shimoikeyana mounted tomb and Kuriyama site were examined using FT-IR microscope and two peaks called amide I and II present in modern silk fibers were compared with those of excavated fibers. It was found that amide ground in a polymer molecule decreased with the progress of degradation and peak intensity of amide group in infrared spectrum decreased correspondingly. The advantages of FT-IR microscopy for the analysis of organic remains in archaeological view point were evaluated and it was concluded that the systematic investigation of spectral change due to the degradation of respective material is essential.

1. Introduction

In the recent archaeological excavations in Japan, several kind of remains consisted of organic substance have been found. Since these remains are usually degraded due to the elapse of long period, the identification of these materials is tedious tasks prior to the conservation treatment. Besides, the analysis of organic materials are generally more difficult than that for inorganic materials such as metal, stone, pottery or glass, because several kind of spectral analysis as well as chromatographic separation process are generally required. Nevertheless, the situation for the analysis of organic materials are ameliorated due to the progress of instrumental analysis as well as several analytical methodology. The development of versatile and rapid analytical procedures is indispensable for the advance of archaeological science.

In this report, the microscopic Fourier

transform infrared spectroscopy(FT-IR) are applied for the identification of fiber materials of excavated textiles. Since the infrared spectrum is characteristic to the molecules constituting the organic materials, infrared spectroscopy is becoming one of the versatile procedures for the rapid distinction of natural fiber into animal, vegetable and mineral constituent. As for textiles or fabrics, fiber materials used for remains are either animal(protein polymer) suchas silk or wool, or vegetable(carbohydrate polymer) such as hemp, ramie, flux or cotton etc..

The results obtained is also useful for the identification of fiber materials depending on fiber morphology or electron scanning microscope, because the degraded state of fibers are well confirmed on the spectral change in infrared spectroscopy.

This report is concerned with the comparative studies on three different samples of silk fibers taken from ancient textiles found at following sites : Fujinoki mounded tomb(second half of sixth century, A.D.), Shimoikeyama mounded tomb(second half of third century, A.D.) and Kuriyama site(the second half of Yayoi-zenki period, A.D.).

To our knowledge, such kind of comparative studies on fiber materials were not yet reported concerning the archaeological remains.

Even the studies on weaving techniques or kind of textiles of ancient period are quite few. Detailed results are published for textiles stored in former Imperial Treasury of Shoso-in(eighth century, A.D.(Nara prefecture)) or of Horyuji temple(seventh century, A.D.(Nara prefecture)).

2. Archaeological significance of textiles studied

It is needless to mention that the studies on fiber materials of ancient period is one of the important subjects in archaeology.

The results of studies on fibers taken from textile fragments of above-mentioned archaeological sites give us significant information about the characteristics of archaeological sites themselves.

The brief summary of archaeological sites mentioned in this report is described in connection with the significance of this study.

Fujinoki mounded tomb is near to Horyuji temple in Nara prefecture. As a result of continuous excavation and investigation from 1985 to 1988 by Archaedogical Research Institute of Kashihara, Nara prefecture, the stone coffin of unidentified person and various kind of mortury goods including variety of textiles(almost of them are of silk) were found.

The archaeologists pointed out the influence of culture in east Asia district on the style and fabrication techniques of most luxurious mortury goods of this tomb.

As far as textile study concerned, the classification of silk fabrics according to the

weaving techniques was published already in detail.

The scientific study on fiber materials or dyestuffs used was, however, roughly done only for a few example until now.

Shimoikeyama mounded tomb(keyhole-shaped) situate at Tenri city in Nara prefecture. The investigation of tomb in 1996 by Archaeological Research Institute of Kashihara, Nara prefecture, revealed the existence of a copper mirror with colored textiles having striped pattern. The results of our investigation clarified that silk fibers used for this textile was kept in good condition. The reason for this is no doubt that the tomb was built with sufficient care to prevent the invation of rain water.

Kuriyama site is at Amagui city in Fukuoka prefecture.

The sample fibers were taken from small fragment of textile(plain weave silk) found at the inside of burial jar in which a corpse supposed to be the head of group was laid. We found that the quality of silk fiber was rather good considering the fabricated period. It is certainly due to the tight seal of jar, preventing the frequent change of humidity during the elapse of long period.

3. Apparatus and Experiments

The infrared spectra were measured using FT-IR microscope(FT 520, Horiba Seisakusho Co., Japan). Trace amount of samples(less than one mg) taken from archaeological remains were made to thin film by pressing on the flat metal plate. The necessary area of sample film was selected on the observation stage of microscope using a mobile aperture under visible light. Only few tens micrometer square of film were usually needed to obtain distinct spectrum of ancient fibers. The observation of spectrum was then performed by switching over the light source to infrared light.

Respective spectral features of silk fibers are shown in Fig. 1(modern silk fibers as reference), Fig. 2(silk fibers of Fujinoki mounded tomb), Fig. 3(silk fibers of Shimoikeyama mounded tomb) and Fig. 4(silk fibers of Kuriyama site).

The infrared spectrum of modern silk fibers(Fig. 1) has two strong absoption peaks at about 1600cm^{-1}, characteristic to the presence of amide group in a polymer molecude. The two peaks are called amide Ⅰ and Ⅱ, respectively. When infrared spectra of ancient silk fibers are compared each other, the decrease in the intensity of amide Ⅰ and Ⅱ peaks seems to be the index for polymer degradation. Two amide peaks finally merge each other in the case of heavily degraded fibers, and the new peaks due to degraded molecules appear instead at about 1000cm^{-1}. Generally speaking, these spectral changes are caused by the progress of polymer degradation due to the elapse of long period. Since the number of amide group in a

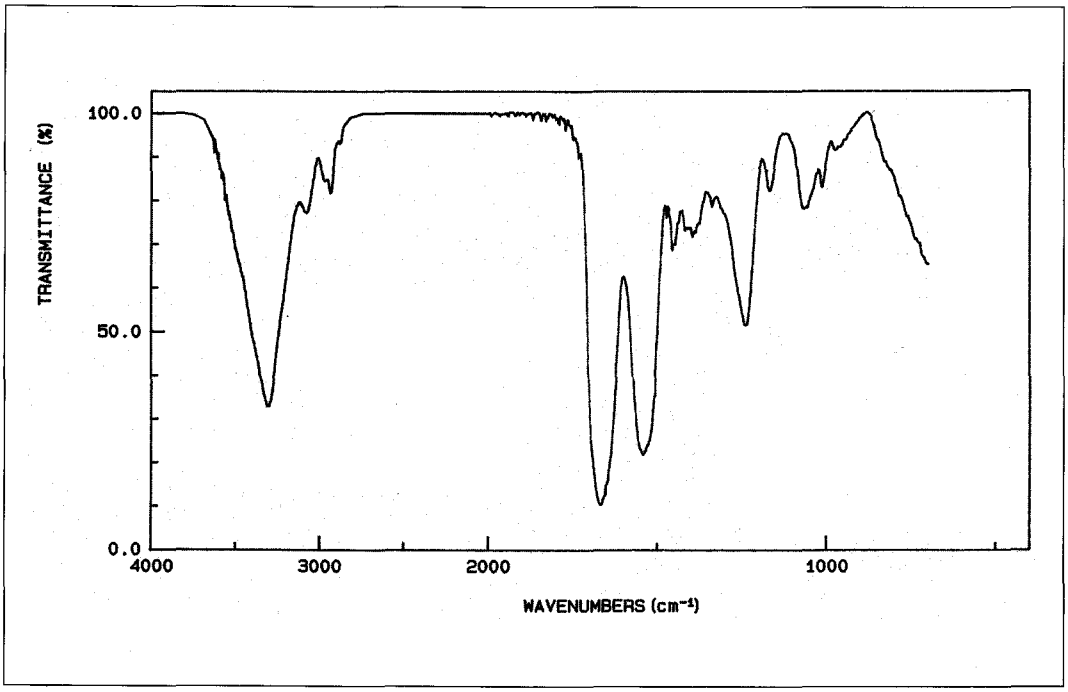

Fig. 1. Infrared Spectra of Modern Silk Fibers Sample

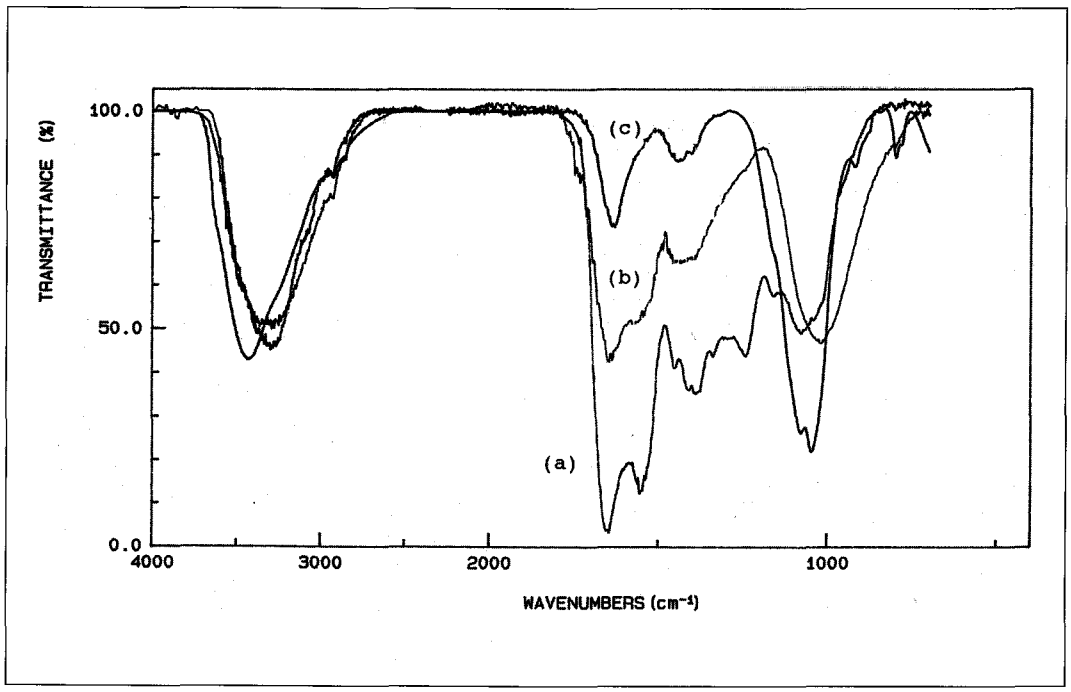

Fig. 2. Infrared Spectra of Silk Fibers from Fujinoki Mounted Tomb:(a)Sample No.22, (b)Sample No.26, (c)Sample No.3

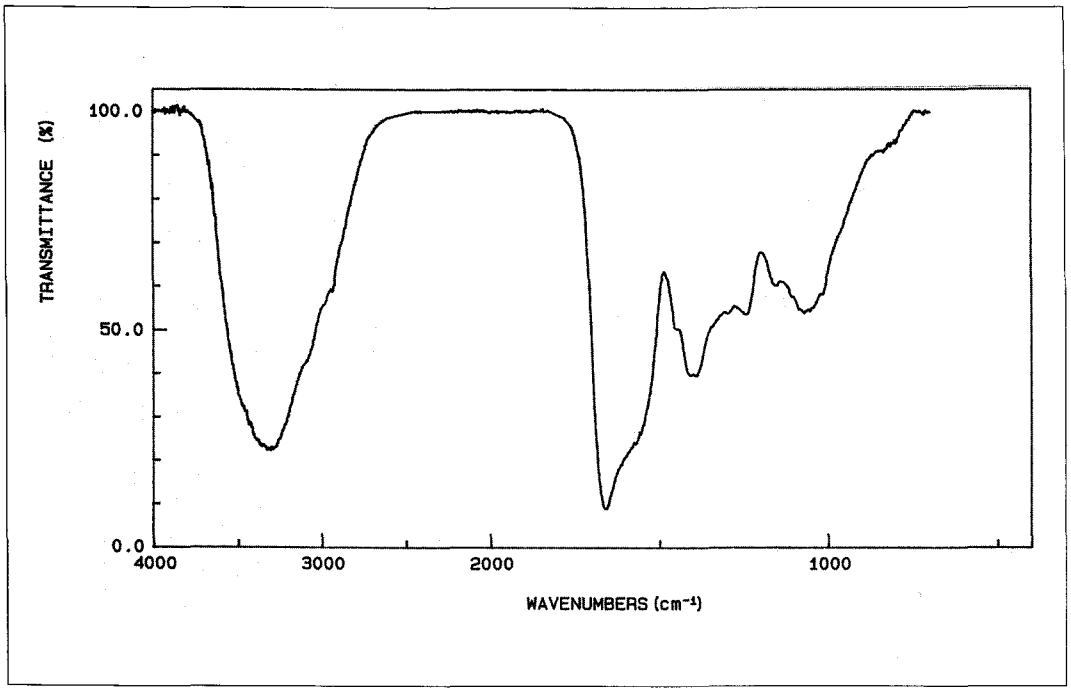

Fig. 3. Infrared Spectrum of Silk Fibers from Fujinoki Mounted Tomb.

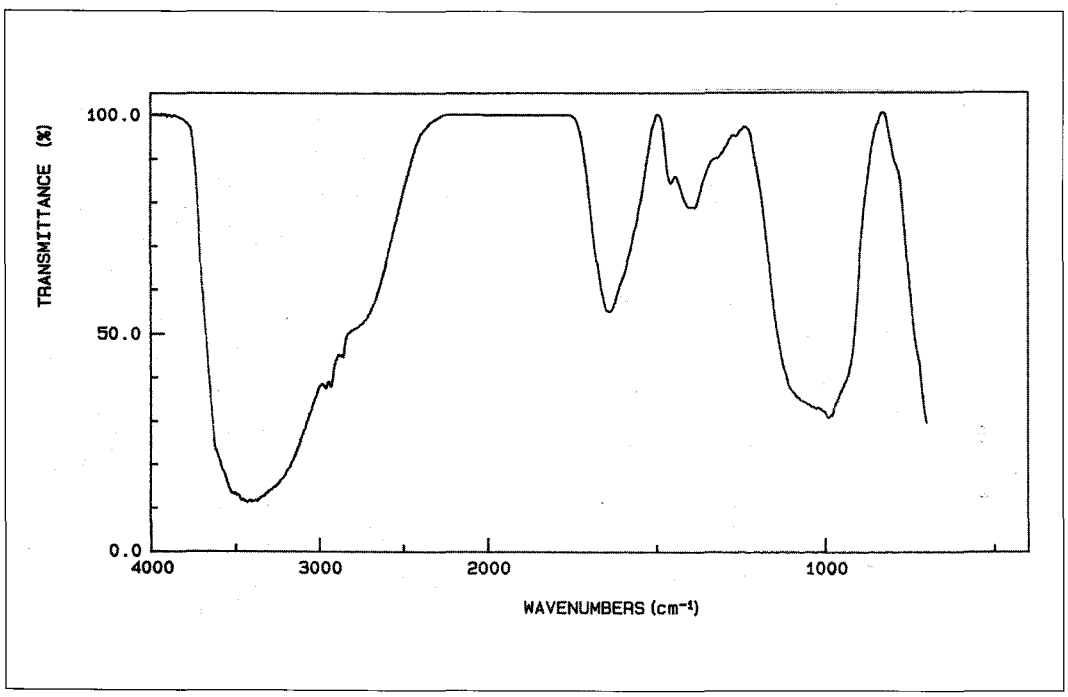

Fig. 4. Infrared Spectrum of Silk Fibers from Kuriyama Site.

polymer molecule decreases with the progress of degradation, peak intensity of amide groups in infrared spectrum decreases correspondingly. Besides, degraded molecule shows new absorption peaks at more low wavenumber region.

It is worth noting that the similar spectral changes are caused by the difference in kept condition even for the remains of the same site(same period) as shown in Fig. 2. The results of the archaeological investigation clarified that the stone coffin of Fujinoki mounded tomb was filled with water several times in the past.

The textiles and fabrics in the stone coffin were maintained in a variety of conditions : a) fibers floated at the surface of water, b) fibers sunk under the water, c) fibers attached at the surface of iron sword or copper mirror(sunk at the bottom of water). Judging by the degree of spectral change, the degraded state is in the order of c) > b) > a). The fibers attached at the surface of iron sword are heavily damaged and the almost organic composition are already lost.

4. Considerations

The advantages of FT-IR microscopy for the analysis of organic remains in archaeological viewpoint are the followings :

1) The method is versatile to distinguish materials as animal, vegetable or mineral.
2) Only trace amount of sample are required. The method is nearly equal to non-destructive analysis.
3) No special and tedious pretreatment are needed prior to the measurement.

On The contrary, organic materials are usually degraded in a various environmental conditions, and according to the elapse of long period. The standard spectral data base are generally useless to identify material without the exact knowledge of spectral change of each material due to the degradation.

When we intend to use FT-IR microscopy as a specific analytical instrument in archaeology, the systematic investigation of spectral change due to the degradation of respective material is essential.