



A novel decomposition technique of friable asbestos by CHClF_2 -decomposed acidic gas

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ABSTRACT

Asbestos was widely used in numerous materials and building products due to their desirable properties. It is, however, well known that asbestos inhalation causes health damage and its inexpensive decomposition technique is necessary to be developed for pollution prevention. We report here an innovative decomposition technique of friable asbestos by acidic gas (HF and HCl) generated from the decomposition of CHClF_2 by the reaction with superheated steam at 800 °C. Chrysotile-asbestos fibers were completely decomposed to sellaite and magnesium silicofluoride hexahydrate by the reaction with CHClF_2 -decomposed acidic gas at 150 °C for 30 min. At high temperatures beyond 400 °C, sellaite and hematite were detected in the decomposed product. In addition, crocidolite containing wastes and amosite containing wastes were decomposed at 500 °C and 600 °C for 30 min, respectively, by CHClF_2 -decomposed acidic gas. The observation of the reaction products by phase-contrast microscopy (PCM) and scanning electron microscopy (SEM) confirmed that the resulting products did not contain any asbestos.

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1. Introduction

Asbestos is the name given to a group of naturally occurring silicate minerals in the amphibole and serpentine series, possessing a typically fibrous-asbestiform habit [1,2]. Amphiboles are double-chain silicates which may assume a fibrous habit being structurally elongated in one preferred crystal direction [1–3]. The general amphibole formula is represented as $\text{AB}_2\text{C}_5\text{T}_8\text{O}_{22}(\text{OH})_2$ and asbestos in the amphibole series (A =empty site, T =Si) has following five kinds: $B=\text{Na}$, $C=(\text{Fe}_3^{2+}\text{Fe}_2^{3+})$ riebeckite; $B=C=\text{Fe}^{2+}$ grunerite; $B=\text{Ca}$, $C=\text{Mg}$ tremolite; $B=\text{Ca}$, $C=(\text{Fe}^{2+},\text{Mg})_5$ actinolite; $B=C=\text{Mg}$ anthophyllite [4]. The commercial term of riebeckite-asbestos and grunerite-asbestos is crocidolite and amosite, respectively. Chrysotile with an ideal chemical formula of $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ belongs to the serpentines which are 1:1 (a tetrahedral and an octahedral sheet) sheet silicates. The curvature of this layer and the tubular morphology of this mineral result from the differences between the bidimensional unit cell dimensions of the two sheets [3].

Asbestos has excellent physical and chemical properties such as high tensile strength, abrasion resistance, heat resistance and

chemical resistance [5]. Therefore, it was used for numerous materials and buildings. It has been, however, clarified that asbestos inhalation causes lung disease such as mesothelioma and asbestosis [6–8]. For this reason, asbestos was banned or limited in USA, Japan and most of the European countries [9], but a large amount of asbestos containing materials exists in our life circumstance. In Japan, it is estimated that the amount of asbestos containing building materials can reach 43 million tons [10]. In the future, these materials will be considered as asbestos containing waste (ACW) which results from renovation or demolition of buildings. Sprayed-on asbestos mixed with cement and products containing a large amount of asbestos are called “friable asbestos”, and molded materials containing a small amount of asbestos such as slates and tiles are called “compact asbestos”. Friable asbestos is very dangerous because of the ease to fly apart into atmosphere when disturbed, so that by statute in Japan friable asbestos must be sorted out as industrial wastes of controlled type and disposed of into controlled landfill type landfill site or inert type sanitary landfill site after melting treatment. But, landfill disposal of friable asbestos never decrease its hazardous nature. Furthermore, the depletion of controlled landfill type landfill site has been a common social problem. The melting treatment is the only allowed method in Japan as an intermediate treatment of friable asbestos, but this method has a big problem that the energy consumption is so large due to the demand of very high operation temperatures at about 1500 °C.

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A number of approaches for decomposition of asbestos and ACW have been reported. The thermal treatment of pure chrysotile results in dehydroxylation at temperatures in the range from 500 °C to 800 °C and subsequent recrystallization at the high temperatures leading to the formation of forsterite and enstatite from Mg-rich and Si-rich regions, respectively [11,12]. Chrysotile is decomposed even at temperatures lower than 500 °C when it is heated for 30 days, a long enough period of time [13]. Thermal decomposition of crocidolite and amosite has been carried out [14,15] and the possibility of recycling of thermally treated or microwave inertised asbestos and ACW is also reported [2,16,17]. The use of microwave energy to inertise ACW has been proved to be competitive in comparison with the conventional treatment in terms of time reduction and energy saving [16,17]. Recent papers revealed that the addition of calcium salts was effective to decrease the decomposition temperatures of asbestos [18,19]. At low temperatures, the calcium salts gave a melt which acted as a flux.

Self-propagating reaction of ACW using the strong exothermic thermite reaction of ferric oxide and magnesium to form magnesium oxide and iron has been reported for the alternative approach to decompose ACW [20]. The mechanochemical method, by which ACW is converted into amorphous materials, is a very simple process [21]. On another front, the decomposition of asbestos has been also attempted in acidic solutions. Mechanisms and pathways of decomposition of chrysotile under static treatment conditions were investigated in fluorosulfonic acid [22]. *In situ* chemical elimination of chrysotile from asbestos containing fireproofing materials was already demonstrated by using the digestion agent containing phosphoric acid, soluble silicofluoride salt and surfactants [23]. Although these dissolution techniques are able to decompose chrysotile in ACW at room temperature, long periods are necessary to digest chrysotile and waste liquid treatment is required.

On the other hand, chlorofluorocarbons (CFCs) and hydrochlorofluorocarbons (HCFCs), which are called “Freon”, are one of the environmentally toxic materials and causes destruction of the ozone layer and global warming. In Japan, the used Freon has a requirement to be decomposed by law not to diffuse in the atmosphere. Freon can be decomposed by the reaction with superheated steam [24], and the decomposition plants of Freon with a continuous pipeline-system by this method have been already marketed [25]. In this method, high reactive acidic gas such as HF and HCl is generated by the decomposition of Freon. We presumed that

asbestos could be decomposed by Freon-decomposed acidic gas, because asbestos has hydroxyl groups and its resistance to acid is relatively weaker than that to base [5].

The technique to decompose asbestos by Freon-decomposed acidic gas has following advantages. Asbestos can be decomposed at low temperatures and in a short time, because Freon-decomposed acidic gas is so strong. Neutralization process of Freon-decomposed acidic gas is reduced by the reaction with asbestos. The waste heat of Freon decomposition is directly used for decomposition of asbestos. Thus, energy consumption of this technique must be very low in comparison with the melting treatment. In this paper, we describe the feasibility of the simultaneous decomposition technique for Freon and asbestos.

2. Experimental

2.1. Apparatus and samples

A schematic illustration of the experimental apparatus is shown in Fig. 1. This apparatus consists of the Freon decomposition plant by superheated steam and the asbestos decomposition reactor tube connected between the Freon reactor chamber and neutralization tank. The apparatus was made of steel with stainless piping units.

Chrysotile-asbestos had been used most widely among the six kinds of asbestos. In Japan, crocidolite and amosite were used as sprayed-on asbestos until prohibition of its use in 1975. Ministry of the environment of Japanese government recently requested us to develop the techniques to decompose ACW containing crocidolite and amosite, as well as chrysotile-asbestos. Thus, following three asbestos samples were used as friable asbestos in this study. Chrysotile-asbestos fibers were purchased from Wako Pure Chemical Industries, and crocidolite containing wastes and amosite containing wastes were collected from lagging materials and fireproofing materials, respectively.

The starting chrysotile fibers contained brucite ($\text{Mg}(\text{OH})_2$) as an impurity. Crocidolite containing wastes mainly consisted of crocidolite over 60 mass% with other crystalline phases of calcite and quartz. Amosite containing wastes contained amosite (30 mass%), chrysotile (3 mass%), calcite and quartz. Incorporation of quartz and calcite must be caused by the additives for production of asbestos materials.

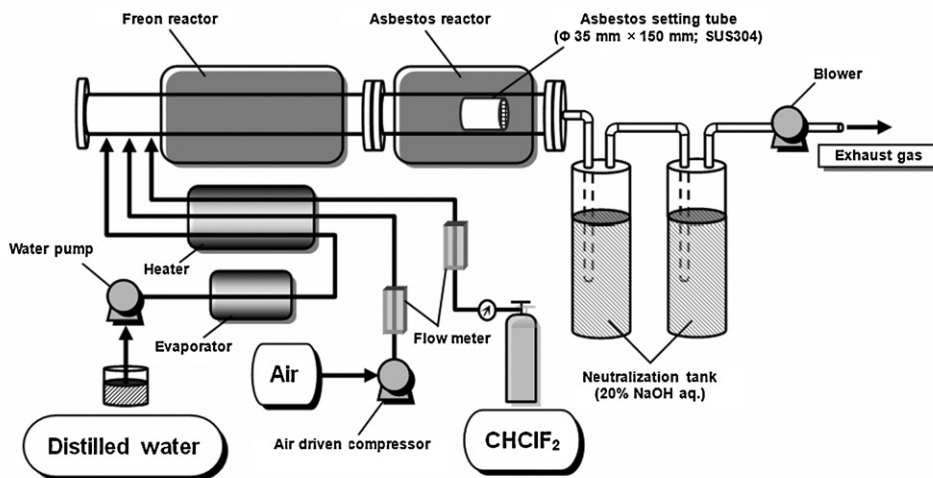
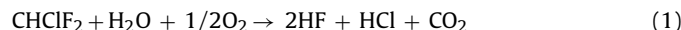


Fig. 1. Schematic illustration of the experimental apparatus.

2.2. Decomposition conditions of CHClF_2

Chlorodifluoromethane (CHClF_2) was used as Freon. The decomposition of CHClF_2 was carried out with the apparatus shown in Fig. 1 under the following conditions: reaction temperature 800°C , and constant flow rates of CHClF_2 , distilled water and air, 3 l/min, 2.43 ml/min, and 8.6 l/min, respectively. Under these conditions, CHClF_2 was completely decomposed with decomposition ratio over 99.9%. The decomposition proceeds by the following proposed chemical reaction to generate acidic gas (HF and HCl) and CO_2 :



2.3. Decomposition of asbestos samples

First, the evaporator, heater and Freon reactor shown in Fig. 1 were heated to 400°C , 600°C and 800°C , respectively. At the same time, the asbestos reactor tube was also heated to a desired temperature from 150 to 800°C . After each temperature reached a proposed one with supplying distilled water and air but not CHClF_2 , an asbestos sample (10 g) in a stainless tube ($\phi 35\text{ mm} \times 150\text{ mm}$) was sealed in the asbestos reactor tube ($\phi 50\text{ mm} \times 500\text{ mm}$). Then, CHClF_2 was immediately supplied for 30 min and the decomposition of CHClF_2 with superheated steam was carried out continuously. After stopped supplying CHClF_2 , the sample was taken out of asbestos reactor tube.

2.4. Analytical methods

After the decomposition process, all the samples were ground for the successive analyses. Powder X-ray diffraction analyses (XRD; Rigaku Rotaflex RAD-RC, radiation Cu $K\alpha$, graphite monochromator, collecting step 0.02° , scanning speed $4^\circ/\text{min}$, $2\theta = 5\text{--}70^\circ$, 40 kV, 100 mA) were carried out on powdered samples to identify crystalline phases before and after the decomposition process. The morphological changes of the samples were observed by scanning electron microscopy (SEM; Hitachi S-530, accelerated voltage 25 kV). A powdered sample (about 30 mg) was dispersed in about 0.8 cm^2 area on an aluminum holder, and two specimens were prepared for each sample. For each specimen, more than 10 fields were observed by changing the magnification. In addition, phase-contrast microscopic observation (PCM; Olympus BX51N-DPH) by using the immersion liquid with $n_D^{25^\circ\text{C}} = 1.550$ and 1.700 (Cargille) was carried out at 400-fold magnification to verify the presence or absence of each asbestos based on Japanese Industrial Standard (JIS A 1481: 2006) [26]. All PCM analyses were conducted on three specimens for each sample. According to the JIS A 1481, the presence of asbestos is defined as the case of more than three fibrous particles of asbestos observed in 3000 particles of three specimens for each sample.

3. Results and discussion

3.1. Decomposition of chrysotile-asbestos fibers

Fig. 2 shows the XRD patterns of the products obtained by the reaction of chrysotile fibers with CHClF_2 -decomposed acidic gas at $150\text{--}800^\circ\text{C}$ for 30 min. Because of inflow of high temperature CHClF_2 -decomposed gas, the lowest controllable temperature of the asbestos reactor tube was 150°C . Nevertheless, chrysotile fibers were completely decomposed and transformed into magnesium silicofluoride hexahydrate ($\text{MgSiF}_6 \cdot 6\text{H}_2\text{O}$) and sellaite (MgF_2) by the reaction with CHClF_2 -decomposed acidic gas at 150°C for 30 min (Fig. 2b). At 200 and 300°C , reaction products contained sellaite and brucite. No silicate compounds were observed (Fig. 2c

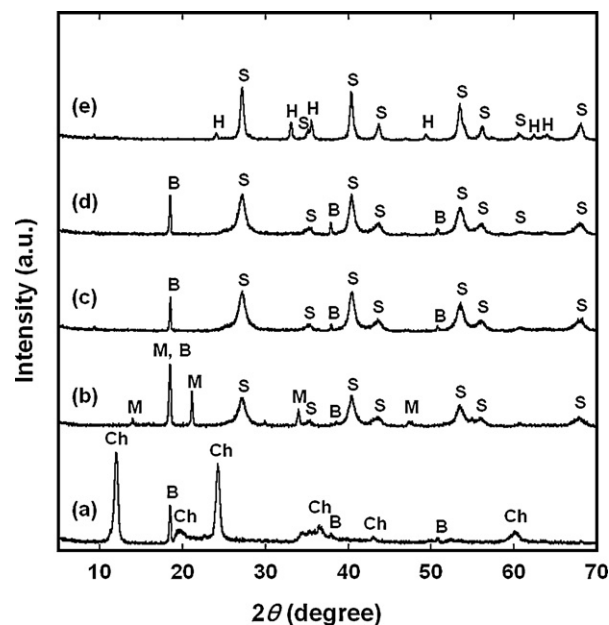
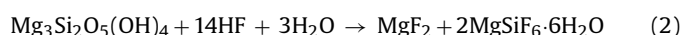


Fig. 2. XRD patterns of (a) raw chrysotile fibers and treated products at (b) 150°C , (c) 200°C , (d) 300°C and (e) 400°C with CHClF_2 -decomposed acidic gas for 30 min. Legend of pattern: B = brucite; Ch = chrysotile; H = hematite; M = magnesium silicofluoride hexahydrate; S = sellaite.

and d). Brucite must be the impurity in the starting chrysotile fibers. At high temperatures, magnesium silicofluoride hexahydrate was decomposed to sellaite and silicon fluoride (SiF_4) [27], and silicon fluoride might be discharged as gas. By the increase in reaction temperature to 400°C , brucite disappeared and sellaite and hematite (Fe_2O_3) were detected in the decomposed product (Fig. 2e). Sellaite must be formed from brucite by the reaction with HF gas. Hematite might be formed by crystallization of iron component included in the structure of starting chrysotile [1,16], but not by contamination from the sample holder because the amount of hematite did not increase even by the reaction at 800°C . Since the thermal decomposition temperature of brucite is in the range of $300\text{--}400^\circ\text{C}$, brucite might stably remain below 300°C . Thus, the ideal reaction of chrysotile fibers with CHClF_2 -decomposed acidic gas can be expressed by following reactions:



Although the decomposition of CHClF_2 with superheated steam generates HCl gas as shown in Eq. (1), chloride compounds were not detected in reaction products. The amount of water is excess for the decomposition of CHClF_2 , so that a small amount of water exists in the decomposition gas. Chloride compounds such as chloromagnesite (MgCl_2) might not be stable under these conditions and replaced by the fluoride compounds. It was confirmed by the separate experiments that hydrophilite (CaCl_2) was formed from portlandite ($\text{Ca}(\text{OH})_2$) at the initial stage of the reaction with CHClF_2 -decomposed gas but replaced by fluorite (CaF_2) as the reaction advanced.

The decomposed products were evaluated by SEM and dispersion staining method using PCM. Fig. 3 shows the PCM and SEM images of the untreated chrysotile fibers and treated products at 150°C for 30 min with CHClF_2 -decomposed acidic gas. PCM analysis with the immersion liquid with $n_D^{25^\circ\text{C}} = 1.550$ shows that chrysotile fibers turn red-purple or blue in color as shown in Fig. 3a. In the treated products, as seen in Fig. 3b, chrysotile fibers were not detected and only fine particles were observed. SEM observation

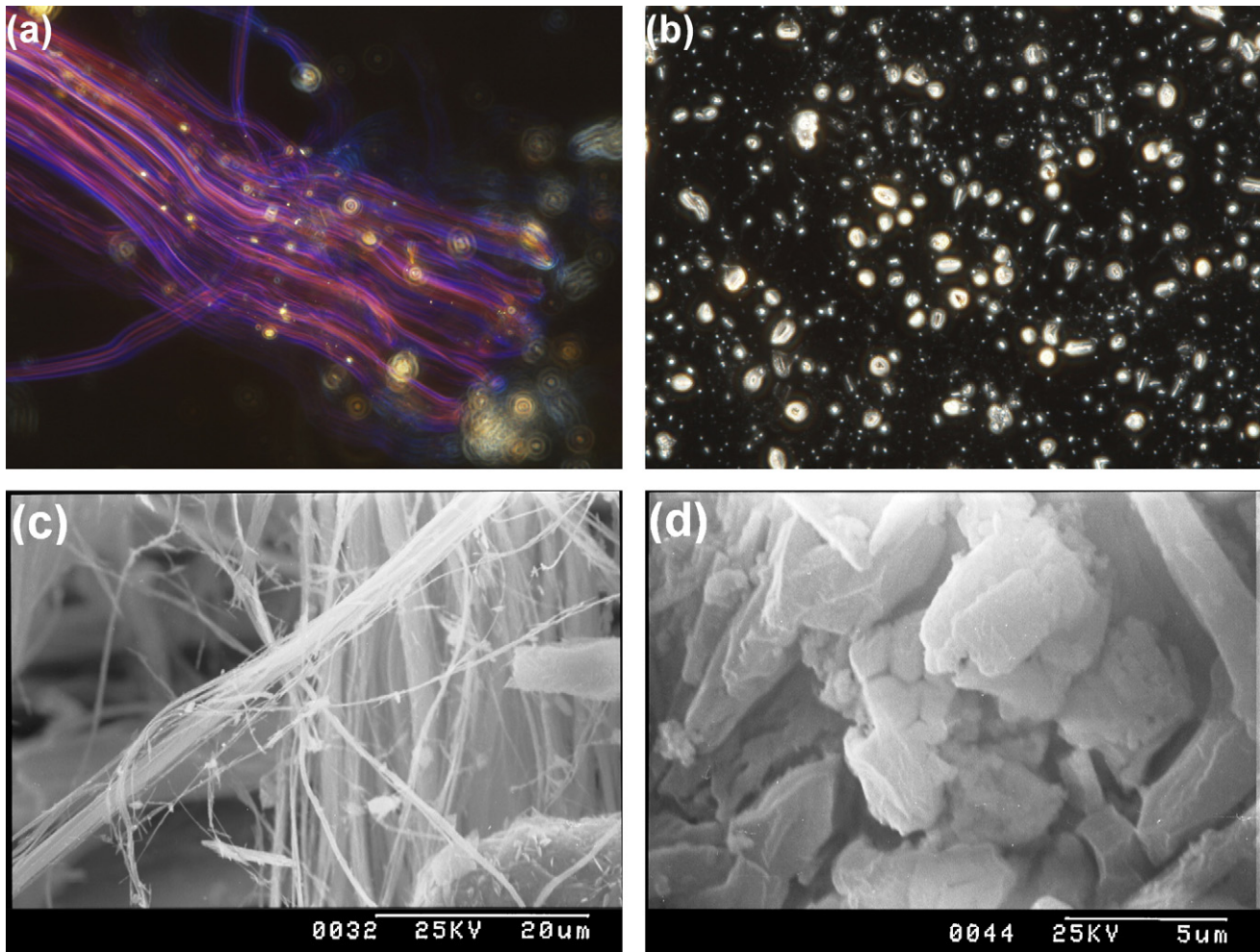


Fig. 3. PCM (a, b) and SEM (c, d) images of before (a, c) and after treatment (b, d) of chrysotile fibers with CHClF_2 -decomposed acidic gas at 150°C for 30 min. (For interpretation of the references to color in the text, the reader is referred to the web version of the article.)

(Fig. 3c and d) clearly shows fibers changed to hard aggregates of fine particles by the reaction with CHClF_2 -decomposed acidic gas.

3.2. Decomposition of crocidolite containing wastes

Fig. 4 shows the XRD patterns of the products obtained by the reaction of crocidolite containing wastes with CHClF_2 -decomposed acidic gas up to 500°C for 30 min. The starting crocidolite containing wastes consisted of crocidolite, quartz and calcite (Fig. 4a). The complete decomposition of crocidolite required 500°C as shown in Fig. 4c. The products at 500°C consisted of hematite, fluorite and quartz. Fluorite was formed from calcite and quartz remained without reactions. Hematite might be formed by oxidation reaction in water vapor from iron component generated through the reaction of crocidolite with CHClF_2 -decomposed acidic gas. On the other hand, sodium and magnesium compounds which must be formed from crocidolite were not detected in the treated products. Sodium and magnesium might form a glass phase.

Fig. 5 shows the PCM and SEM images of the untreated crocidolite containing wastes and treated products at 500°C for 30 min with CHClF_2 -decomposed acidic gas. As shown in Fig. 5a, crocidolite turns blue in color by using the immersion liquid with $n_D^{25^\circ\text{C}} = 1.700$. In the treated product shown in Fig. 5b for PCM analysis, any blue colored fibers were not detected. The SEM analyses before (Fig. 5c) and after the treatment (Fig. 5d) shows that crocidolite

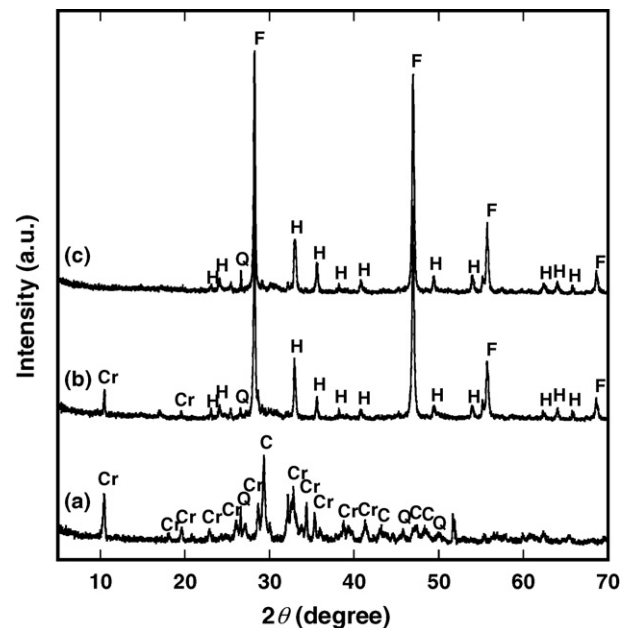


Fig. 4. XRD patterns of (a) as-received crocidolite containing wastes and treated products at (b) 450°C and (c) 500°C with CHClF_2 -decomposed acidic gas for 30 min. Legend of pattern: C = calcite; Cr = crocidolite; F = fluorite; H = hematite; Q = quartz.

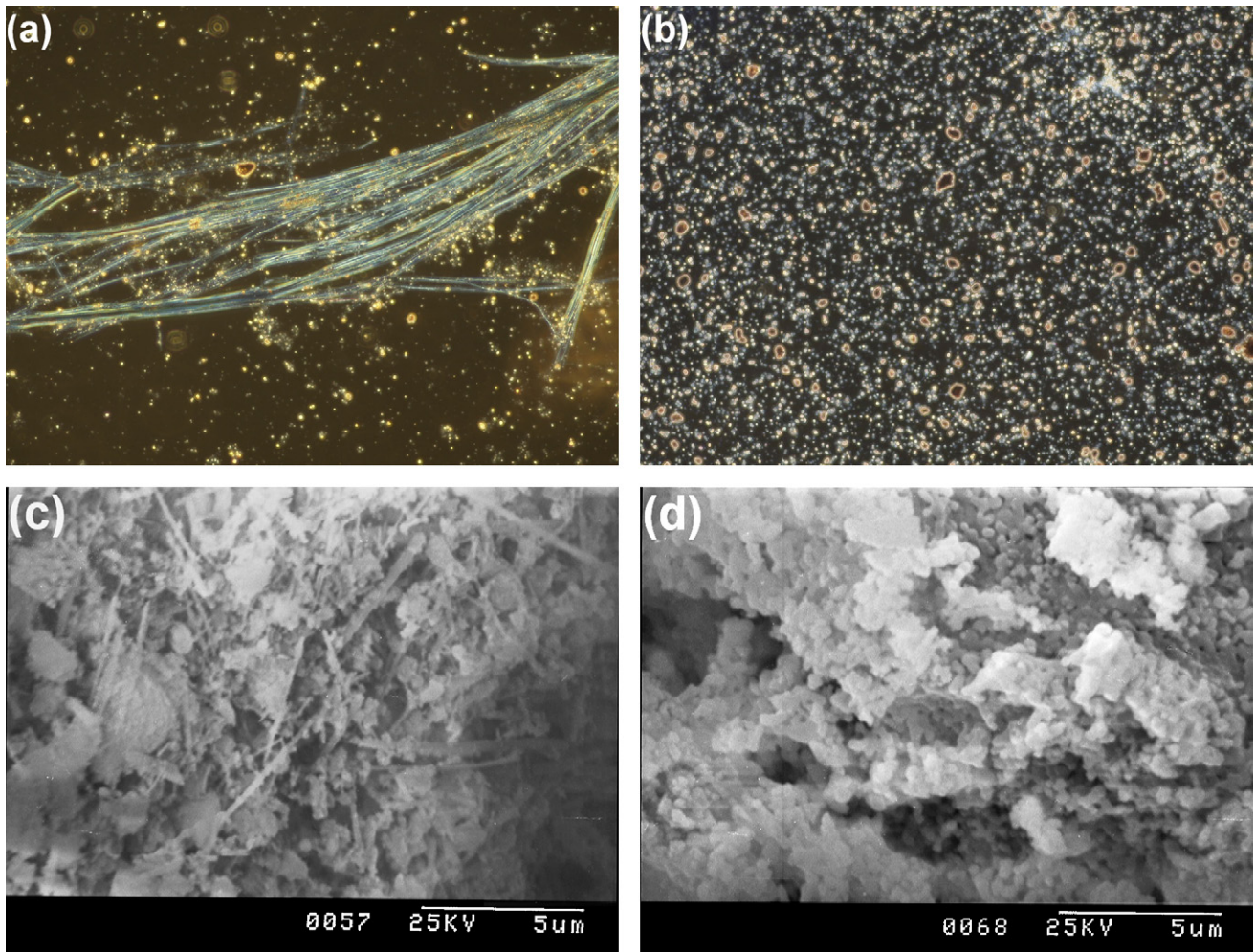


Fig. 5. PCM (a, b) and SEM (c, d) images of before (a, c) and after treatment (b, d) of crocidolite containing wastes with CHClF_2 -decomposed acidic gas at 500 °C for 30 min. (For interpretation of the references to color in the text, the reader is referred to the web version of the article.)

lite fibers disappeared in the sample. These results suggest that the treated product was no longer defined as crocidolite containing.

3.3. Decomposition of amosite containing wastes

Fig. 6 shows the XRD patterns of the products obtained by the reaction of amosite containing wastes with CHClF_2 -decomposed acidic gas up to 600 °C for 30 min. The starting amosite containing wastes consisted of amosite, chrysotile, quartz and calcite (Fig. 6a). Chrysotile in the samples was already decomposed at temperatures below 550 °C (Fig. 6b). Amosite containing wastes were almost decomposed at 600 °C for 30 min by CHClF_2 -decomposed acidic gas, though a tiny diffraction peak which may correspond to the major diffraction peak of amosite was detected in the treated products as shown in the inserted figure of Fig. 6c. The decomposition products of amosite containing wastes at 600 °C were similar to those of crocidolite containing wastes. Hematite was the common product, because iron is the main constituent of both amosite and crocidolite. In the case of amosite containing wastes, a small amount of sellaite was detected in the treated products. The amount of sellaite must be increased in the treated products, because amosite containing wastes include chrysotile and magnesium content of amosite is larger than that of crocidolite. Weberite ($\text{Na}_2\text{MgAlF}_7$) and calcium aluminum fluoride (CaAlF_5) must be formed by the reaction of as-received amosite containing wastes and acidic gas.

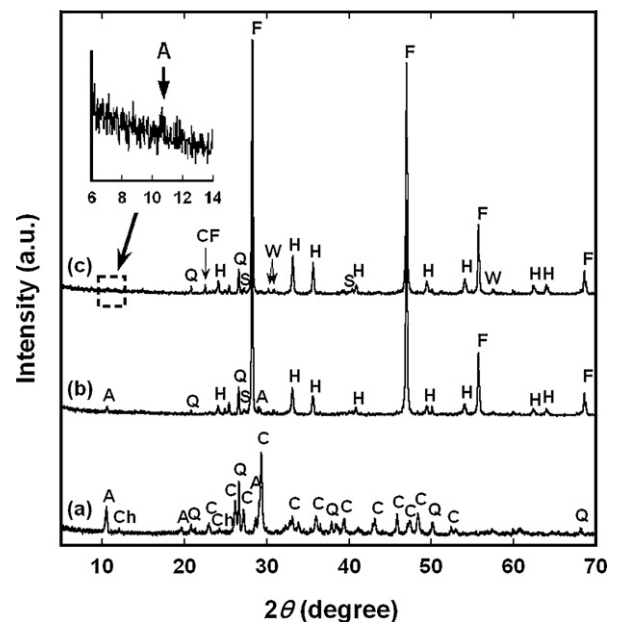


Fig. 6. XRD patterns of (a) as-received amosite containing wastes and treated products at (b) 550 °C and (c) 600 °C with CHClF_2 -decomposed acidic gas for 30 min. Legend of pattern: A = amosite; C = calcite; CF = calcium aluminum fluoride; Ch = chrysotile; F = fluorite; H = hematite; Q = quartz; S = sellaite; W = Weberite.

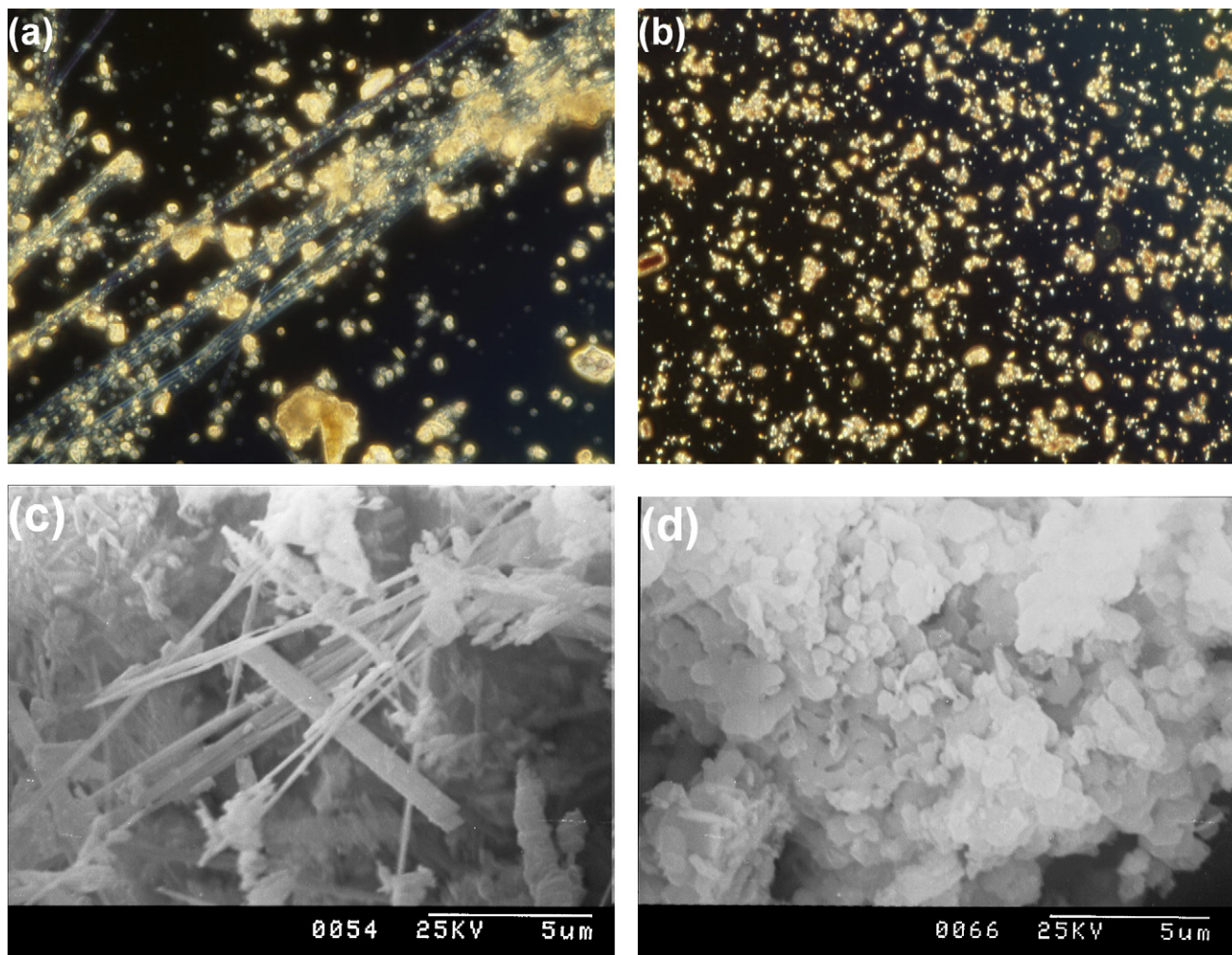


Fig. 7. PCM (a, b) and SEM (c, d) images of before (a, c) and after treatment (b, d) of amosite containing wastes with CHClF_2 -decomposed acidic gas at 600°C for 30 min. (For interpretation of the references to color in the text, the reader is referred to the web version of the article.)

Fig. 7 shows the PCM and SEM images of the untreated amosite containing wastes and treated products at 600°C for 30 min with CHClF_2 -decomposed acidic gas. For PCM analysis with the immersion liquid with, amosite turns blue in color as shown in Fig. 7a. In the $n_D^{25^\circ\text{C}} = 1.700$ treated products, colored amosite fibers were not detected as shown Fig. 7b. Based on Japanese Industrial Standard (JIS A 1481: 2006), it is concluded that amosite was decomposed by the reaction with acidic gas at 600°C . SEM observation revealed that needle-like crystals of amosite in the starting sample as shown in Fig. 7c changed to fine particles (Fig. 7d) by the reaction with CHClF_2 -decomposed acidic gas.

The decomposition of crocidolite and amosite in ACW required at high temperatures in comparison with that of chrysotile fibers because crocidolite and amosite are more resistant to acids than chrysotile [5]. However, asbestos of chrysotile, crocidolite and amosite was completely decomposed by the reaction with CHClF_2 -decomposed acidic gas at much lower temperatures than the melting treatment.

4. Conclusions

A new decomposition technique of friable asbestos has been developed by using acidic gas generated by the decomposition of CHClF_2 with superheated steam. Asbestos of chrysotile, crocidolite and amosite was decomposed by the reaction of CHClF_2 -

decomposed acidic gas at much lower temperatures than the traditional melting method, that is, at 150°C , 500°C and 600°C for 30 min, respectively. Freon and friable asbestos were simultaneously decomposed by this technique.

According to Eqs. (1) and (2), 1 mole of CHClF_2 can ideally decompose 1/7 mole of chrysotile. In our experiment, CHClF_2 gas of 90 l (4.017 mole) was used to decompose 10 g of chrysotile (3.609×10^{-2} mole), which suggests that 6.29% of the decomposed gas was effectively used to decompose chrysotile. This low efficiency is caused by placing a very small amount of the chrysotile sample in the large reaction tube. The efficiency would be increased by filling the chrysotile sample in the reaction tube and selecting the optimized reaction time.

The real decomposition plant of Freon can decompose 25 kg of CHClF_2 in an hour, and 30 plants are now in operation in Japan. If all plants work for 8 h in a day and 200 days in a year, CHClF_2 of 1200 tons and chrysotile of 549 tons can be ideally decomposed in a year. In 2004, friable asbestos of 17,019 tons was disposed of into controlled landfill type landfill site and that of 1315 tons was treated by the melting method in Japan. The capacity to decompose friable asbestos by the technique using Freon-decomposed acidic gas is very small in comparison with the total amount of friable asbestos waste. Even so, we expect that the technique using Freon-decomposed acidic gas contributes to treatment of friable asbestos waste, because friable asbestos is decomposed with low energy

consumption by this technique in comparison with the melting method. Furthermore, acidic gas produced by Freon decomposition plants is effectively used and the amount of waste after the decomposition of ACW is comparable to that produced by neutralization process of Freon-decomposed acidic gas. This method needs only modification of the existing Freon decomposing plants, which may be another advantage.

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