Development of switchable mirror glass

— R&D strategy toward its practical use —

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"Switchable mirror" is a new thin film material that can be switched between transparent and mirror states. Using this material on window glass saves energy by effectively shading rooms from sunshine, thereby decreasing cooling load in summer. In this paper, we introduce our R&D strategy for further development and practical deployment of this material. In addition to R&D of the material itself we also measured the amount of energy saved when the material was used in the windows of buildings. The results we obtained from such field tests will enable us to develop a window glazing with better energy-saving performance.

Keywords : Smart window, energy efficiency, cooling load, chromogenic material, durability

1 Introduction

The Materials Research Institute for Sustainable Development is engaging in research for "energy-saving building components" that are materials useful in reducing the CO_2 emission in the civilian sector. The Energy Control Thin Film Group is in charge of the research of windowpane glass. The percentage of energy consumed in cooling and heating reaches about 30 % of the energy consumption in the civilian sector, and the window is a component that greatly affects cooling and heating efficiency. While the objective of a window is to let in light, ordinary windowpane allows the permeation of heat as well as visible light, and it is a factor that leads to the loss of insulation of the building. Increasing the insulation property of the window has great energy saving effect, and recently, the uses of highly insulating pair glass and low-e glass are becoming common. In Japan where the summers are very hot, the energy-saving effect can be further increased by effectively blocking the sunlight (shading) in addition to increasing insulation. The switchable glass is a windowpane that can automatically control the inflow and outflow of light and heat.

The switchable glass is fabricated by coating the glass with thin film material that can reversibly vary the optical property (chromogenic material). There are several types of switchable glasses. For example, an electrochromic glass accomplishes the switching electrically,[1] a thermochromic glass changes according to temperature,[2] and a gaschromic glass changes by surrounding atmosphere (gas).[3] Among these switchable glasses, there is a long history of research of the electrochromic glass, and some types of glasses are already available on market overseas.

In Japan, the electrochromic switchable glass research was conducted by major glass manufacturers until the 1990s. One of the authors (Yoshimura) was involved in the research of switchable material from the days of the National Industrial Research Institute of Nagoya that was the precursor of AIST Chubu. The switchable material was investigated as one of the passive energy-saving techniques in the national project of 'Sunshine Project' and 'New Sunshine Project'. Although a product with excellent performance was fabricated, the research was terminated at around 2000 because the prospect for practical use could not be conceived due to cost issues. The research entered the so-called "valley of death" or the "period of nightmare."

At that time, the author (Yoshimura) started to look at the "switchable mirror" that might reignite the research of this switchable glass. The switchable glass was a new switchable material discovered in the Netherlands in 1996.[4] While the conventional switchable material changed from transparent to deep blue color, the new material changed from transparent to a mirror state. In the mirror state, it reflected and cut off the sunlight. Because the mirror state reflected and cut off more sunlight compared to conventional switchable glass, this allowed a window with higher shading performance. In conducting the switchable mirror thin film research, we had in mind the achievement of the practical use of the energy-saving windowpane from the beginning, not just doing research for materials. In this paper, we present how the research was conducted to achieve practical use of the switchable mirror glass.

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2 Issues in achieving the practical use of the switchable mirror

When the research for the switchable mirror was started at AIST in 2001, the following issues were identified as points that must be solved, and researches were started: (1) the improvement of optical property, (2) the improvement of durability, and (3) the development of an electrochromic switchable mirror.

2.1 Improvement of optical property

Figure 1(a) shows the basic structure of the switchable mirror composed of the magnesium alloy thin film as the switching layer, that is coated with a thin catalyst layer. In an as-deposited state after film deposition, the magnesium alloy layer is in a mirror state since it is metallic, and when it is exposed to an atmosphere containing diluted hydrogen, the switching layer becomes hydrogenated by the action of palladium catalysts, and the layer turns into an insulator and turns transparent. Next, when this is exposed to an atmosphere containing oxygen, the hydrogen in the hydride reacts with oxygen by the action of palladium catalysts and gets pulled out as H_2O, and the film returns to a metallic state and becomes a mirror. The switching mirror state where the mirror and transparent states switch back and forth was first found in the thin films of rare earth metals such as yttrium and lanthanum, and several new materials were found later (Table 1). The alloy of magnesium and transition metal was found in the United States in 2001, and this was considered to be a better material to be applied to large glass, because it was less expensive and was thought to have higher durability compared to the rare earth metals. AIST focused on this material and started research for the first time in Japan. However, when this material was first discovered, the transmittance of visible light was about 20 % in its transparent state, and the color was dark brown that was not suitable for practical use. AIST started research on increasing the visible light transmittance in the transparent state of the switching mirror, and found that the visible light transmittance in the transparent state can be increased to about 50 % by using the Mg rich Mg-Ni alloy thin film, as shown in Fig. 1(b).[13] This was a big step forward to practical use.

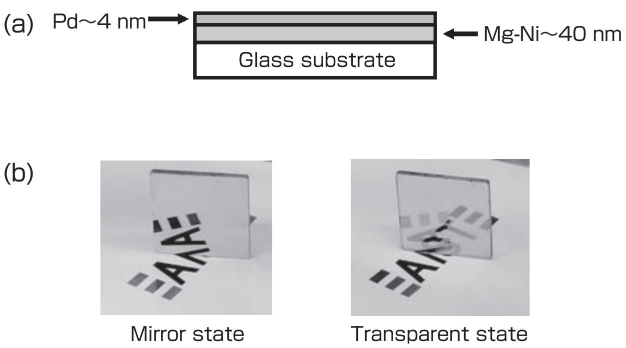

Fig. 1 (a) Basic structure of the switchable mirror, and (b) the magnesium-nickel (Pd/Mg_6Ni) switchable mirror thin film with excellent optical property developed at AIST

Table 1. Types of switchable mirror thin film materials

First generation	Rare earth metal Y[4],La[4],etc. Vrije Universiteit, Amsterdam 1996
Second generation	Rare earth/magnesium alloy Gd-Mg[5],Sm-Mg[6],Y-Mg[7],etc. Phillips 1997
Third generation	Magnesium/transition metal alloy Mg-Ni[8],Mg-Ti[9],Mg-Co[10],etc. Lawrence Berkeley Laboratory 2001
Fourth generation	Magnesium/alkaline earth metal alloy Mg-Ca[11],Mg-Ba[12],Mg-Sr[12] AIST 2009

However, when the Mg-Ni alloy thin film is used as the switching mirror layer, the sample is slightly brown in the transparent state although it has excellent switching property. This color is not considered preferable for use in the windows of buildings and vehicles. Therefore, we conducted research for improving the color neutrality in the transparent state, and found that almost coloreless transparency can be obtained in cases of hydrogenation by using the Mg-Ti alloy[14] or Mg-Nb alloy[15] as the switching layer. However, although the Mg-Ti alloy thin film or Mg-Nb alloy thin film is colorless in the transparent state, the visible light transmittance is inferior compared to the Mg-Ni alloy thin film. Our research group recently found that we could obtain colorlessness during hydrogenation and increase the visible light transmittance by using the thin films of Mg-Ca,[11] Mg-Ba, and Mg-Sr[12] alloys. Figure 2 shows the optical transmittance spectra in the transparent state of each material. The materials of magnesium and alkaline-earth metal can be called the fourth generation materials made originally by AIST, unlike the

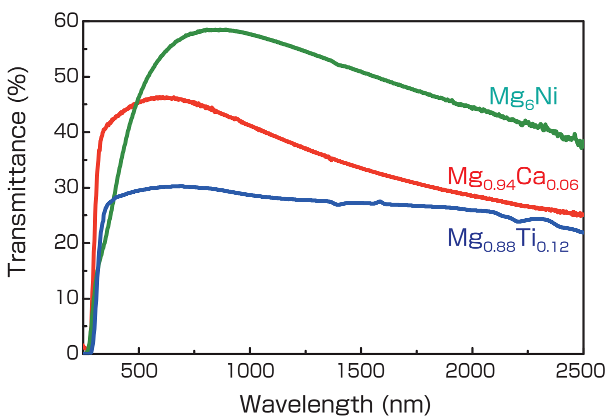

Fig. 2 Comparison of the transmission spectra in the transparent state of the magnesium-nickel (Mg_6Ni) switchable mirror, magnesium-titanium ($Mg_{0.88}Ti_{0.12}$) switchable mirror, and magnesium-calcium ($Mg_{0.94}Ca_{0.06}$) switchable mirror

conventional switchable mirror thin film category. Their practical use is expected.

At what level the variation range of the transmittance and reflectivity is set depends on the use. For example, for use in the windshield of a car, visible light transmittance of 70 % or above in the transparent state is necessary, and this condition has not been cleared at present. To raise the transmittance to this level, it is necessary to optically design the whole multilayer thin film including the palladium and protective film layers, not just the switchable layer composed of magnesium alloy thin film. We are currently engaging in this development.

2.2 Improvement of durability

For the switchable mirror thin film material, a product with fairly practical performance has been achieved in terms of its optical property. However, the greatest problem of this material is the rapid deterioration due to repeated switching. For example, Fig. 3(a) shows the variation in the optical transmittance at wavelength of 670 nm when switching is repeated using hydrogen gas diluted to 4 % using argon, for the Pd/Mg_4Ni thin film. It can be seen that the optical variation range gradually decreases with repeated switching, and the deterioration progresses rapidly particularly after 140 times.[16] In the practical use of the switchable mirror, the improvement of durability is the most important issue, and we have made various attempts to improve this.

Deterioration occurs due to a combination of several factors, and one factor is that the Mg in the Mg-Ni thin film migrates to the surface through the Pd layer, due to the repetition of hydrogenation and dehydrogenation. We found that the migration of Mg can be controlled by inserting a metal thin film of Ti or others as a buffer layer between the Pd and Mg-Ni layers.[17] As seen in Fig. 3(b), the deterioration does not occur up to about 400 times when this buffer layer is inserted. However, rapid deterioration occurs in this sample also. This is due to the change in volume due to the hydrogenation and dehydrogenation, and this damages the Pd layer. To prevent this, a certain type of protective film is useful. For example, when the Teflon (PTFE) protective film was vapor-deposited on the surface of the sample to which the Ti buffer layer is inserted, the switching could be repeated up to about 1,500 times (Fig. 3(c)).[18] Yet for practical use, durability of about 10,000 times is necessary, and we are continuing the research for further improvement of durability.

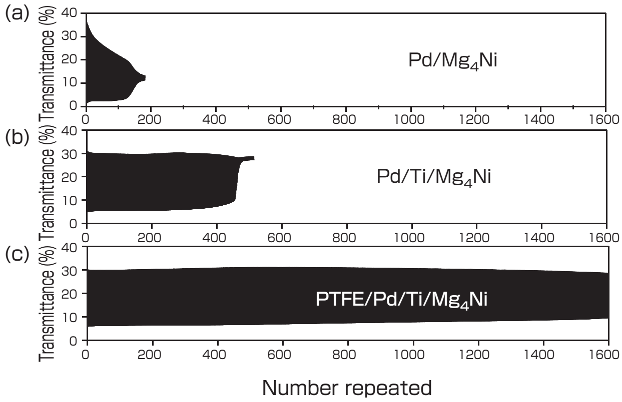

Fig. 3 Comparison of switching durability
(a) Switchable mirror thin film with magnesium alloy layer and palladium layer only, (b) switchable mirror thin film with inserted buffer layer, and (c) switchable mirror thin film with buffer layer and protective film coating (Teflon layer).

2.3 Realization of the electrochromic switchable mirror

To switch the switchable mirror by a gaschromic method, it is necessary to use the double layer (pair) glass. Since this may be difficult depending on the use, electric switching may become necessary in some cases. For switching by an electrochromic method, we initially studied a device using an alkaline water solution. However in a device that uses a solution, the magnesium dissolves when the positive charge is applied to the switchable thin film side and short-circuiting was the only way to return from the transparent to the mirror state, and this was not practical. Therefore we developed an all-solid-state electrochromic switchable mirror that did not use the solution state.[19]

Figure 4 shows the structure of the all-solid-state switchable mirror device that is being fabricated presently.[20] It is a multilayer structure in which the transparent conductive film (ITO), tungsten oxide thin film, tantalum oxide thin film, Al thin film, Pd thin film, and Mg-Ni alloy thin film are stacked. The films are fabricated by the magnetron sputtering method. The tungsten oxide thin film is a layer for storing the hydrogen ion, tantalum oxide thin film is the electrolyte layer, Pd thin film is the layer for promoting the passing of hydrogen, and the Mg-Ni alloy thin film is the switchable layer that switches from the mirror to the transparent state. The Al thin film is the buffering layer that prevents the Pd from dispersing into the tantalum oxide layer through repeated switching. The state of

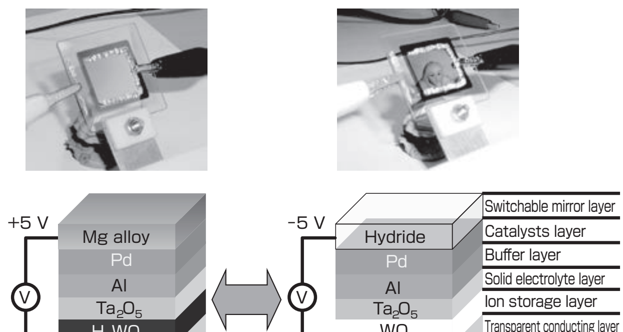

Fig. 4 Photograph and structure of the all-solid-state electrochromic switchable mirror device

switching in the fabricated device (about 3 cm on each side) is shown in Fig. 4. When -5 V voltage is applied to the switchable mirror thin film side, the Mg-Ni layer becomes hydrogenated as the hydrogen ion inside the tungsten oxide thin film transfers, and the mirror state changes to the transparent state in about 20 seconds. When +5 V is applied, the hydrogen ion drops out of the Mg-Ni layer and transfers into the tungsten oxide thin film, and the transparent state changes to the mirror state in about 15 seconds.

The switchable mirror device is characterized by the fact that any substrate can be used for film forming. Therefore, if a transparent plastic substrate is used instead of a glass substrate, a bendable switchable mirror film can be fabricated. The plastic device where switching can be done at the same level as the glass substrate has been successfully developed.[21] If the switchable mirror film can be realized, the switchable mirror property can be achieved by simply covering the existing glass with it, and the range of application will expand significantly.

For the gaschromic switchable mirror, a sample with a large surface area can be fabricated easily by a large sputtering machine, and switching can be accomplished in about 20 seconds even if it is of a meter size. However, for the electrochromic switchable mirror, the switching speed declines rapidly as the size increases, and initially, a piece of glass of about 15 cm square took about one hour to switch. We worked to increase the switching speed using various methods, and currently, switching can be accomplished in about 30 seconds for the 15 cm square sample. Further breakthrough is necessary to realize an electrochromic switchable mirror of larger sizes, and this is being studied as the most important topic for the electrochromic switchable mirror.

3 Research scenario for achieving practical use of the switchable mirror glass

To achieve practical use of the switchable mirror glass, joint research with glass manufacturers is mandatory. However, as mentioned in the “Introduction,” the Japanese glass manufacturers terminated the research for switchable glass at around 2000, and it was necessary to raise the awareness of the glass manufacturers that this can be done practically and it is worth restarting the R&D for switchable glass. Therefore, we thought it was important to speed up solving the unsolved problems at that moment, and to show how much energy-saving performance would increase when this glass was used.

The Materials Research Institute for Sustainable Development not only studies the materials, but also has a dedicated building to measure the energy-saving performance when such materials are used. Figure 5 is the photograph of the Testing Facility of Energy Performance that is built on the compounds of the AIST Chubu in Nagoya. The third floor of this building is divided into small rooms each with a size of about 2.5 m on one side. There is a single window on the south side of each room, and two windowpanes can be installed. Each room is equipped with the same air conditioning device, and the power consumption of the air conditioning can be monitored individually. By installing various types of windowpanes in each room, the load on the air conditioning for each room when it is set at a certain temperature can be compared. As described in the following chapter, the switchable mirror glass that can be installed in a building was fabricated, the energy-saving capacity was evaluated and compared with the conventional energy-saving glass, and the major reduction on the cooling load was demonstrated. By conducting measurements in this environment, important findings were obtained that could not be obtained if the research was limited to studying materials only. For example, it was found that the orientation of the window was very important with the energy-saving glass and that it is necessary to carefully consider the entry of sunlight. By feeding back such findings to the materials research, the development of glass with higher energy-saving performance becomes possible.

On the other hand, the switchable mirror thin film has various uses other than for building glass. Figure 6 shows the major applications. In achieving practical use of these materials,

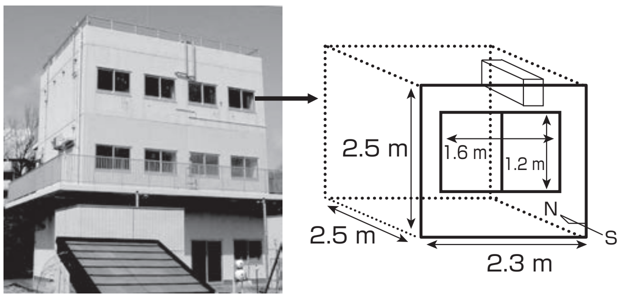

Fig. 5 Environment Friendly Experiment Building

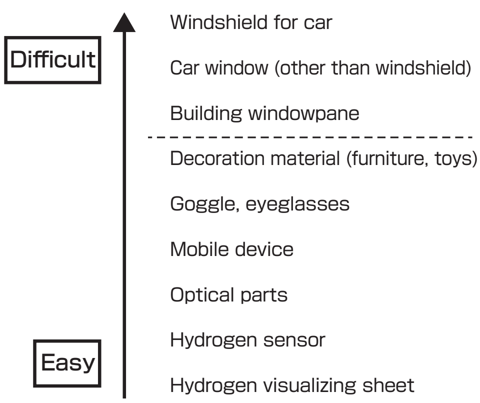

Fig. 6 Application of the switchable mirror thin film

there are cases that can be relatively easily commercialized and those that are quite difficult. While we are ultimately aiming to achieve practical use of the switchable mirror for windows of buildings and vehicles, our strategy is to create commercial products starting from those that are possible, in the process of reaching our final goal.

For example, the switchable mirror thin film undergoes optical change when it comes into contact with an atmosphere containing hydrogen, and therefore, hydrogen can be detected by observing this optical change.[22] The conventional hydrogen sensors must be heated, and the sensor may become an ignition source in cases where the hydrogen leaks. The hydrogen sensor that uses the switchable mirror thin film reacts with hydrogen at room temperature so there is no need of heating. Also, the switchable mirror thin film deposited on the tip of the optical fiber can be used as the sensor, and the reflectivity variation can be used for monitoring using the fiber end. This allows the hydrogen sensor to be of no danger of becoming an ignition source, as there will be no electrical circuit on the detector part. Also, as a unique usage of this material, it can be used as a “hydrogen visualizing sheet” where the presence of hydrogen can be checked visually as with the litmus paper. Since this could be relatively easily put to practice, it was commercialized by our joint researcher Atsumitec Co., Ltd., and the product has become available on the market from 2010. From the last fiscal year, joint research has been started for use in relatively small-scale applications in specific products.

4 Demonstration of energy-saving performance

Among the applications of the switchable mirror thin film, the use for which there is greatest expectation is for the energy-saving windowpane. However, the degree to which energy-saving performance could be obtained when the switchable mirror glass is used was unknown. Therefore, we fabricated a large switchable mirror glass that can actually be installed in a building, and conducted the measurements of energy-saving performance.[23]

For the windowpane that can be installed in a building, we fabricated the switchable mirror glass with the structure shown in Fig. 7(a). Pair glass consisting of two panes of transparent glass of 5 mm thickness with 8 mm gap in between was prepared, and the switchable mirror thin film was deposited using Mg_4Ni thin film as the switchable layer on the inner side of the glass on the outside of the room or building. The size when mounted on the aluminum sash was 1.2 m in height and 0.8 m in width. Figures 7(b) and (c) are photographs of the mirror and transparent states of the fabricated switchable mirror windowpane. This is the view of the switchable mirror window from outside the building. It was almost a perfect mirror in the mirror state, and the interior of the room could not be seen. When it was switched to the transparent state, the window on the opposite side could be seen through the room, and it was like a transparent glass. Another characteristic of the switchable mirror glass is that, in the mirror state, the view outside can be seen when the person inside the room gazes outside from the window.

The fabricated full-size switchable mirror windowpane was installed in the Testing Facility of Energy Performance seen in Fig. 5, and the measurements of the cooling load were taken. Figure 8 shows the measurements taken at the end of August. Before the measurement, the same transparent double glasses were installed in two rooms, and the temperature was set to 28 °C to check that the cooling loads were the same. Next, the window of one room was changed to the switchable mirror glass, switched to the mirror state, and the cooling load power was measured. On that day, the cooling load of the room with the transparent glass window was 1,065 Wh, while the cooling load of the room with the switchable mirror window in the mirror state was 720 Wh, and about 34 % energy savings was accomplished. It was demonstrated that when the switchable mirror windowpane in the mirror state was used, there was a significant reduction of the cooling load particularly on days with high solar radiation.

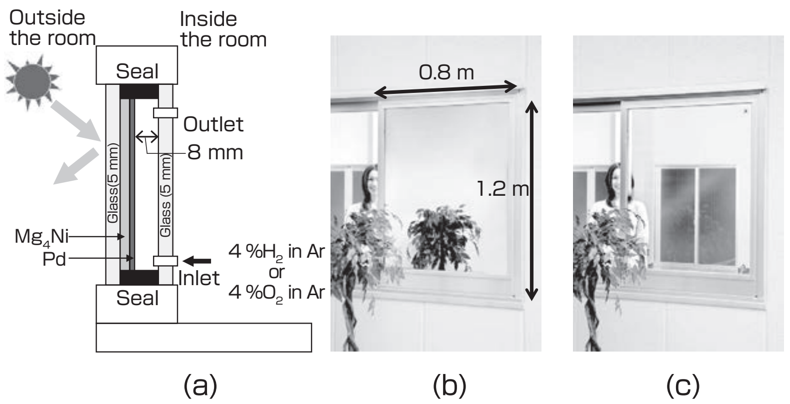

Fig. 7 Switchable mirror windowpane of actual size
(a) Structure, (b) mirror state, and (c) transparent state

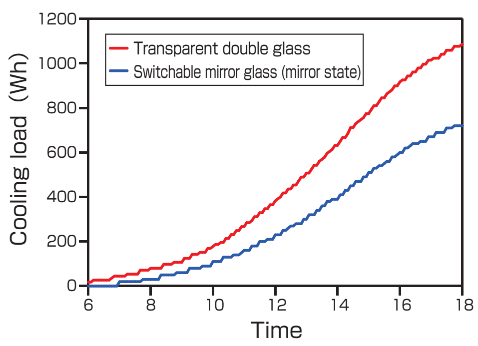

Fig. 8 Comparison of the cooling load using windows fit with transparent double glass and switchable mirror glass

5 Conclusion

We have been conducting research to achieve practical use of the switchable mirror for about ten years. Some applications that were relatively easy to put into practice have been commercialized. In the research for using the switchable mirror thin film in the energy-saving windowpane, we not only conduct the R&D for materials, but also conduct the measurements of energy-saving effect when it was actually used in a building. By reflecting the result in the material development, we are conducting research to realize a windowpane with higher energy-saving performance.

According to the experiment results obtained so far, the cooling load reduction using the switchable mirror windowpane is greater than any other energy-saving windowpane reported so far, and we hope to contribute to the CO_2 reduction in the civilian sector, by achieving practical use of this product as soon as possible.

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Discussions with Reviewers

1 Process of discovering the Mg-Ca, Mg-Ba, and Mg-Sr switchable mirror materials

Question (Norimitsu Murayama, Advanced Manufacturing Research Institute, AIST)

Your discovery that Mg-Ca, Mg-Ba, and Mg-Sr have excellent properties as switchable mirror materials is wonderful. Please describe the process by which you made this discovery. Did you evaluate the properties by making certain projections? Or did you come across them through serendipity?

Answer (Yasusei Yamada)

Another research group at the Materials Research Institute for Sustainable Development was conducting research to make magnesium non-flammable, as this metal was difficult to use due to its highly ignitable property. This research had a totally different objective from our research, and it was discovered that magnesium becomes less flammable by adding calcium. In the switchable mirror, it is necessary to control the oxidation of magnesium to increase durability. Knowing the result of that group, we thought if magnesium becomes less flammable by adding calcium, perhaps it would also become less prone to oxidation. That was the start in studying this material. We fabricated the material, took measurements, and found that it had excellent optical property, and this led to the discovery of the new material. In general, calcium is thought to be a highly active, unstable metal, and if we were not at the Materials Research Institute for Sustainable Development, we wouldn't have had the opportunity to learn about fire-resistant magnesium, because that is research in a different field, and we would never have thought of adding calcium to magnesium that is also highly active.

2 Intellectual property strategy for the Mg-Ca, Mg-Ba, and Mg-Sr switchable mirror materials

Question (Norimitsu Murayama)

For obtaining the intellectual property rights for the Mg-Ca, Mg-Ba, and Mg-Sr switchable mirror materials, can you tell us what were your strategies and what problems you faced, as much as you can disclose?

Answer (Yasusei Yamada)

In Japan, our research group owns the patents for the basic materials of the switchable mirror. However, in Europe and the US, the patent for the thin films of alloys of magnesium and almost all transition metals have been taken. To escape this limitation, we applied for the patent for the switchable mirror material using the thin films of alloys of magnesium and alkaline-earth metals. In this case, we carefully studied why the addition of the alkaline-earth metal was more effective compared to the conventional transition metals, and rather than giving the phenomenological description of the effect, we claimed the superiority of the material from a logical perspective. By doing so, we stated that the magnesium alloy thin film with added alkaline-earth metal was a totally new category of material for the switchable mirror. Whenever a patent for a basic material is taken, the patent may not be accepted if it is merely improvement done on the structure of the device that uses that material. Therefore, significance of being able to file the patent as our original basic material is great.

3 Novelty of all-solid-state switchable mirror device

Question (Norimitsu Murayama)

Is the all-solid-state switchable mirror device the authors' idea?

Answer (Kazuki Yoshimura)

Although the all-solid-state device had been developed for the conventional electrochromic material that uses tungsten oxide thin film as the switchable layer, the switchable mirror using the magnesium alloy thin film had not been realized. Initially, the device was fabricated by layering the switchable, electrolyte, and counter electrode layers according to the structure of conventional all-solid-state switchable electrochromic element, but the device did not work. As a result of reviewing why it did not function, we thought that the layers should be reversed for the switchable mirror. We created a device with the reverse structure, and were able to develop a device with a good switching function. This reversed multi-layer thin film structure is our original idea.

4 Organization of the issues for achieving practical use of the switchable mirror glass

Question (Kazuo Igarashi, Institute of National Colleges of Technology, Japan)

As the three issues in achieving the practical use of the switchable mirror glass, you mention the improvement of optical property, the improvement of durability, and the development of an electrochromic method. While the former two are essential issues that must be cleared, the third does not seem to be a requirement since it depends on the use. What are your thoughts on this?

Answer (Kazuki Yoshimura)

Since the usage of a gaschromic method is very limited, switching by an electrochromic method is important in achieving practical use. In 2002, we had no idea whether switching could be done by an electrochromic method for the magnesium alloy thin film, and it was an important point in the practical use research of the switchable mirror.

5 Positioning of the upsizing technology in achieving practical use of the switchable mirror glass

Question (Kazuo Igarashi)

In upsizing from the laboratory level to actual use level, it can be expected that new issues will arise such as optical property or durability. Therefore, I think upsizing was a major issue in practical use, but it is not mentioned in this paper. How should I consider the positioning of upsizing in the practical use strategy?

Answer (Kazuki Tajima)

Upsizing can be done relatively easily for the gaschromic method, while it is a problem for the electrochromic method. I added some description on this point.

6 Durability of the switchable mirror

Question (Norimitsu Murayama)

You mention that the durability for repeated switching in achieving practical use of the switchable mirror is currently about 1,500 times. What is the prospect of extending the durability to about 10,000 times?

Answer (Yasusei Yamada)

For durability, there was a major progress recently, and we are finding out that we may be able to develop a switchable mirror material with hardly any deterioration. By developing this technology, we have the prospect of creating a material with durability of about 10,000 times within this fiscal year.

7 Efforts for achieving practical use of the switchable mirror glass

Question (Norimitsu Murayama)

You quantitatively demonstrated the energy-saving effect of the switchable mirror glass at the Environment Friendly Experiment Building. As a result, how did the way the industry look at switchable mirror glass change? Also, do you have any plans such as setting up some corporate consortium as the scenario for achieving practical use of the switchable mirror glass?

Answer (Kazuki Yoshimura)

For the switchable mirror, the people of the glass manufacturers have been paying attention to it since 2003 as a new switchable material. However, due to the historical progress as described in this paper, they decided to sit and wait to see whether this material could actually be put to use. In such a situation, fabricating the switchable mirror windowpane that can be actually installed in a building and demonstrating the large cooling load reduction effect compared to other energy-saving glass materials were regarded with great enthusiasm. The glass manufacturers are specifically considering joint research.

To take the switchable mirror glass to the stage of an actual product that can be used as the windowpane for buildings, the developments of parts other than the switchable mirror thin film, such as the switching system that can be used in actual windowpanes and its power supply system are necessary. Currently, the research is being continued, and the prospect for practical use is good if these issues are solved. At that point, we plan to make powerful appeals to the glass manufacturers and accelerate the practical use research by perhaps forming a consortium.