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Challenges in the Practical Application of Dismantlable Adhesives

Introduction

In recent years, increasing emphasis has been placed on reusing end-of-life products and utilizing recycled materials. Progress is being made towards building concrete systems in this area, particularly in the European Union (EU), which has implemented measures such as the Ecodesign for Sustainable Products Regulation (ESPR). Rather than doing so of their own volition, companies are being legally required to focus on designs that facilitate disassembly and ease of material separation, meaning such elements can be considered prerequisites for future business activities. Against this backdrop, ThreeBond is part of several domestic consortia in Japan and is engaged in efforts to develop and implement technology to produce sealants and adhesives with properties that facilitate disassembly, in consideration of societal changes. In this article, the author introduces dismantlable sealants and adhesives currently in development and describes the challenges encountered when looking to their application in material recycling and remanufacturing.

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1. Background of Development of Dismantlable Sealants and Adhesives

In addition to the rising demand for raw materials as exemplified by rare metals and growing supply chain risks due to factors such as the concentration of supplies in certain regions, waste processing challenges and efforts to achieve carbon neutrality have led to the widespread permeation of circular economy-based approaches.[1, 2] Moving forward, while regional differences and the setting of transition periods are foreseeable, it is increasingly likely that products that do not consider circular economies, namely products that do not incorporate consideration of resource circulation from the design stage, will face restricted access to markets. Furthermore, in the resource recovery and recycling industries, factors such as advanced separation technology and efficient resource recovery mechanisms can be considered key differentiating factors.

Adhesive bonding enables low cost, lightweight bonding of surfaces, and can be used to bond different materials together. However, the presence of adhesives in products can impede post-collection recycling processes. Methods such as removing bonded sections by hand before starting crushing and pulverizing processes require labor, which can increase recycling costs. The presence of sections bonded with adhesives can also increase the manual labor required when repairing and reusing products that contain such bonds, leading to higher maintenance costs.

In consideration of these factors, development is being carried out to create dismantlable adhesives that maintain performance during use but can have their adhesive strength reduced with a specific external stimulus (trigger), enabling the bond to be separated.

2. Separation of Adhesive Bonds with Oxidizing Agent-induced Dismantlable Adhesives and Consideration of Material Recycling and Remanufacturing

This section describes ThreeBond's in-development oxidizing agent-induced dismantlable adhesive, which is a dismantlable adhesive that uses artificial oxidizing agents such as sodium hypochlorite as the trigger. This description starts with a description of the material characteristics, followed by current challenges and proposed work process solutions, before concluding with testing the bond quality when adhesive is removed from a bonded item, and the item is subsequently bonded once more.

2-1 Oxidizing Agent-induced Dismantlable Adhesives

In the design of dismantlable adhesives, ThreeBond is focusing on dismantling mechanisms that use acylhydrazine oxidation decomposition (Figure 1).[2]

Acylhydrazine has been shown to be oxidized and decomposed by sodium hypochlorite, which is a known oxidizing agent.[3] The oxidizing agent-induced dismantlable adhesive, which has this acylhydrazine structure in its network polymer after curing, is notable in that its trigger, sodium hypochlorite, is a chemical not found in nature. Compared to heat and light, which are naturally-occurring triggers, the risk of unintentionally reducing the adhesive bond strength is low. Furthermore, aqueous sodium hypochlorite solutions include bleaching agents used in homes, making them cheaper than substances such as organic solvents. They also do not pose a risk of catching fire and are not subject to legal restrictions regarding storage or use. Aqueous sodium hypochlorite solutions can be considered easy-to-handle chemicals as immersion

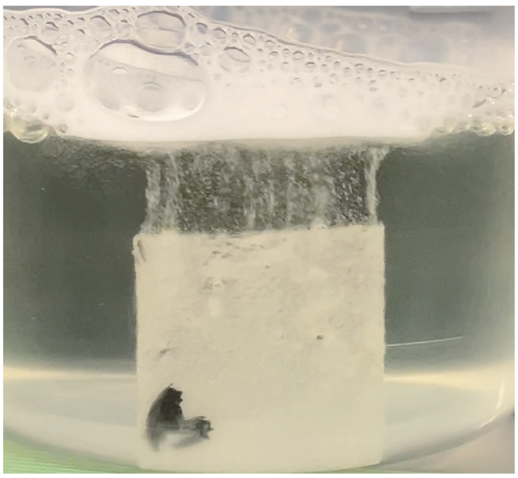


Fig. 2 Release of Gas When Cured Oxidizing Agent-induced Dismantlable Adhesive is Immersed in Aqueous Sodium Hypochlorite Solution

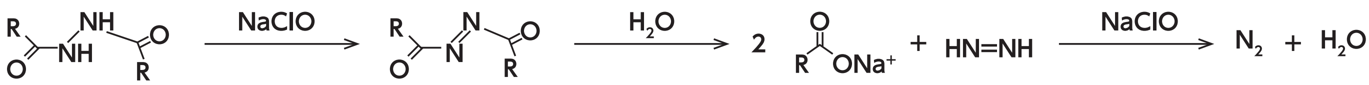


Fig. 1 Acylhydrazine Oxidative Decomposition Reaction

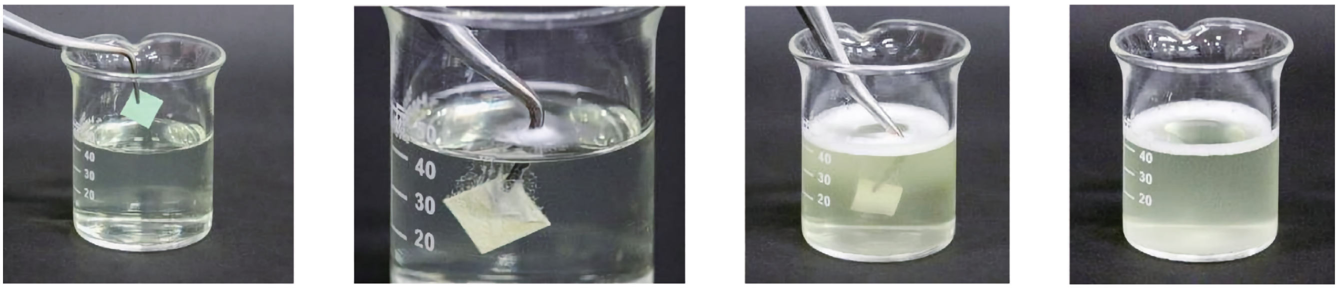


Fig. 3 Decomposition When Cured Oxidizing Agent-induced Dismantlable Adhesive is Immersed in Aqueous Sodium Hypochlorite Solution

liquids for dismantling. Based on these factors, development of adhesives that can be dismantled with oxidizing agents such as sodium hypochlorite is being carried out.

As shown in Figure 2, in the decomposition of the cured adhesive, immediately after immersion, nitogen gas is released and the decomposition reaction progresses. If the cured material is approximately 10 × 10 × 0.5 mm in size, it dissolves in the aqueous sodium hypochlorite solution (6% concentration) within a few hours (Figure 3).

In the decomposition of cured bonding agents, sodium hypochlorite decomposes each part that it comes into contact with in turn, starting from the contact surface, and the bonding agent dissolves in the water. Next, as a new surface is revealed, the decomposition process is expected to repeat, progressively decomposing the adhesive.

In this way, the adhesive is dissolved in the aqueous sodium hypochlorite solution. In other words, if this adhesive is used to bond plastic and similar parts, the cured adhesive could be removed from the adherend, making it possible to recycle plastic.

2-2 Recycling Methods and Challenges with Using Oxidizing Agent-induced Dismantlable Adhesives

As previously described, oxidizing agent-induced dismantlable adhesives dissolve in aqueous sodium hypochlorite solutions. While it initially appears to be a fast chemical reaction that achieves efficient decomposition, because adhesives are typically used for bonding, the surface area that comes into contact with the aqueous sodium hypochlorite solution is limited. Consequently, if an object is immersed in an aqueous sodium hypochlorite solution while bonded, the time needed to dismantle the adhesive bonds is several times longer than when the adhesive is immersed as a mass. To investigate the speed of decomposition when bonded, square glass films with sides of 30 mm and a thickness of 70 µm were bonded together across their entire surfaces. The adhesive was then cured and the films were immersed in an aqueous sodium hypochlorite solution. As shown in Figure 4, which plots the length of adhesive that has dissolved against time, the time taken for about 3 mm to dissolve was 8 hours. The slow dismantling rate presents an issue, and examinations of ways to increase the speed are ongoing.

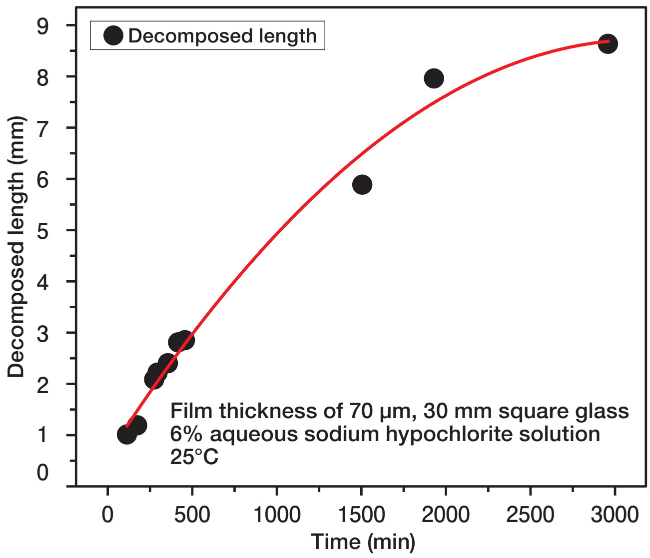


Fig. 4 Relationship Between Decomposed Length and Time for Oxidizing Agent-induced Dismantlable Adhesives

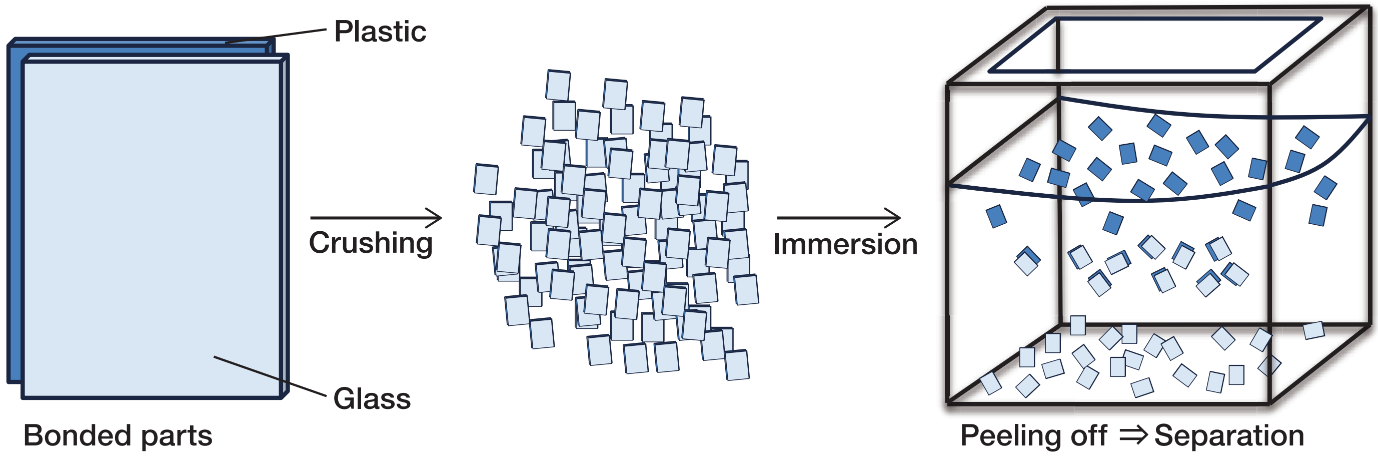


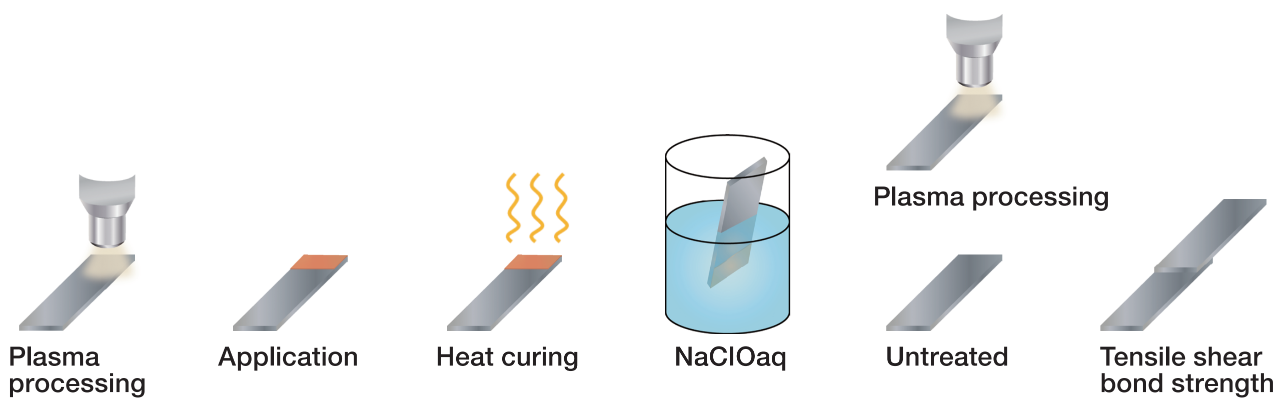
Fig. 5 Envisaged Material Recycling Method Combining Crushing with Oxidizing Agent-induced Dismantlable Adhesives

At the same time, even with this slow decomposition speed, combining this method with crushing of bonded components may bring the material recycling processes to fruition. The envisaged process is shown in Figure 5. For example, if parts where glass and plastic have been bonded can be crushed well, this fine crushing increases the exposed surface area of the adhesive layer, facilitating contact with the aqueous sodium hypochlorite solution. This can reduce the time required for decomposition. Furthermore, when the glass and plastic peel apart during immersion, it is anticipated that the difference in specific gravity can be used to separate the materials at the same time.

2-3 Testing of Bond Quality During Recombination of Parts Using Oxidizing Agent-induced Dismantlable Adhesives

Next, if the adhesive is exposed, as is the case with fillet bonding, the aqueous sodium hypochlorite solution can gain ample access. It is possible that this could also be used in adhesive bonds designed for remanufacturing, meaning the dismantling and reassembly of collected components. However, the question of whether adhesion as strong as the initial bond can be achieved is an issue to address. In particular, consideration must be given to the possibility that changes have occurred in the condition of the peeled surface.

To that end, the following test was performed with polyphenylene sulfide (PPS) as the adherend. The test process is shown in Figure 6. First, plasma processing was performed on the PPS bonding surface. Next, the adhesive was applied and heat curing was performed, then the cured adhesive was immersed in an aqueous sodium hypochlorite solution and peeled off. Plasma processing was performed on the peeled surface once more, then readhesion was performed. At each stage, contact angle measurement (for surface free energy calculation) and tensile shear bond strength measurement were performed on the adherend to evaluate the effects of each process.



Adherend: PPS; plasma processing: Nihon Plasmatreat Inc., 5 mm, 100 mm/sec; Curing conditions: 150°C × 30 min

Fig. 6 Experiment Workflow for Adherend Reuse Test (Oxidizing Agent-induced Dismantlable Adhesives)

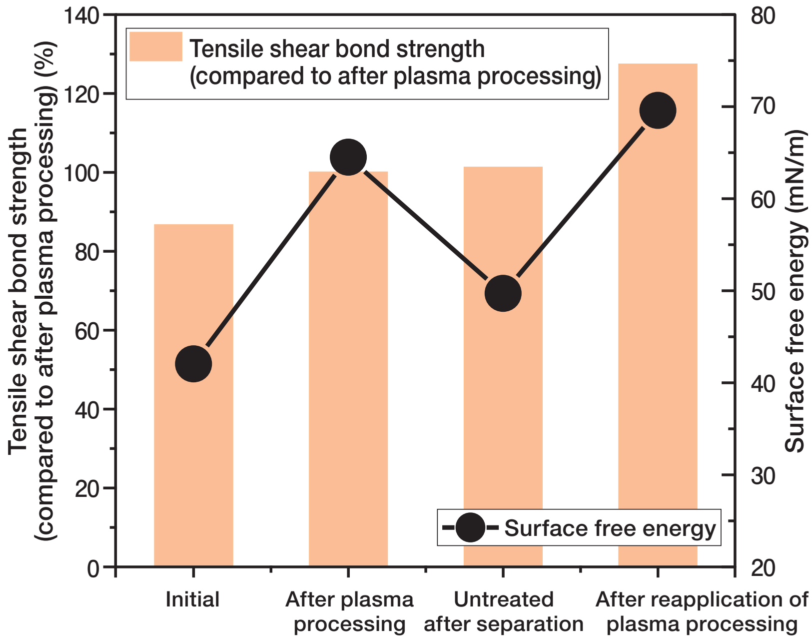


Fig. 7 Tensile Shear Bond Strength and Surface Free Energy Before and After Readhesion (Oxidizing Agent-induced Dismantlable Adhesives)

The tensile shear bond strength and surface free energy measurement results at each stage are shown in Figure 7. For the tensile shear bond strength, the first tensile shear bond strength result after plasma processing was set as 100%, and the strength at other stages is expressed as a percentage of this value. The surface free energy was calculated based on the contact angle between the water and the diiodomethane, using the OWRK method.

Plasma processing can be expected to have three effects (Figure 8). The first is removal of foreign matter from the surface. The second is generating polar functional groups believed to contribute to adhesion. The third is creating surface irregularities that provide an anchoring effect. Plasma processing is widely used, particularly with engineering plastics, because these effects enable stable adhesive bonding.

The changes in tensile shear bond strength and surface free energy values shown in Figure 7 can be considered in the following way. The change from the initial state to the state after plasma processing is due to the plasma processing effects described above. It can be said that, because the emission of plasma modifies the PPS surface, the surface free energy increases, which in turn increases the bond strength. Next, regarding the change from after plasma processing to after separation, while the bond strength does not change, the surface free energy decreases to near the initial value. At this point, the applied adhesive had dissolved in the aqueous sodium hypochlorite solution and there was no adhesive residue. The decrease in surface free energy may be due to the fact that the polar functional groups generated through the plasma processing were buried in the bulk. In one study into the effects of plasma processing, it was observed that the polar

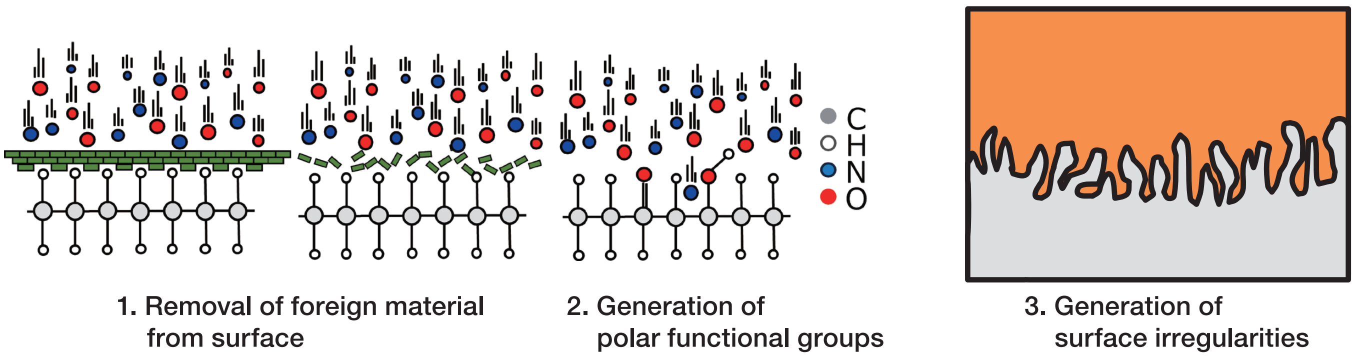


Fig. 8 Expected Effects of Plasma Processing

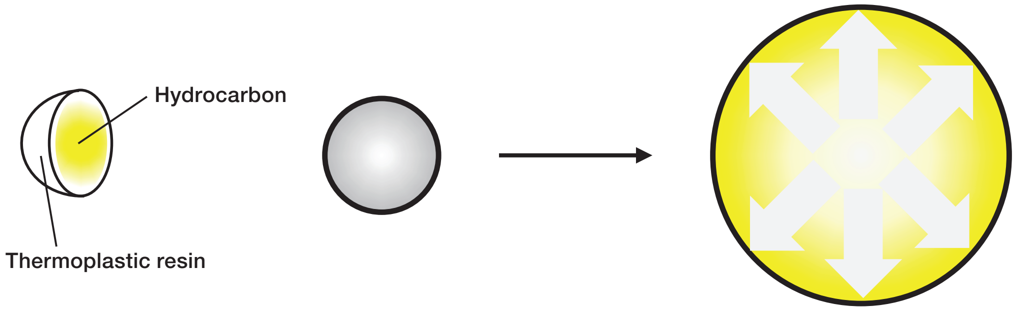


Fig. 9 Example of Thermal Expansion Capsules

functional groups generated on a polypropylene surface disappeared over time.[4)] As was the case in that other study, it is assumed that the cause with PPS was also that the polar functional groups generated due to plasma processing entered the inside of the bulk. However, it is also assumed that the fact that PPS surface irregularities formed due to plasma processing do not disappear, even after adhesive curing and adhesive bond separation, meant that there was no reduction in bond strength due to the anchor effect. Furthermore, in terms of the change from after separation to after reapplication of plasma processing, both surface free energy and bond strength increased. This is believed to be because the bond strength increased based on the total number of times that plasma processing was performed.

Compared to the initial PPS state, the measured surface free energy and bond strength after plasma processing had been performed twice were both higher than after plasma processing had been performed once and were close to the values after the reapplication of plasma processing.

This suggests that incorporating plasma processing into the separation and readhesion process for plastics such as PPS makes it possible to achieve stable adhesive bonds, even with readhesion.

As described earlier in this article, in addition to adhesive design, consideration of quality challenges related to material recycling and remanufacturing is commencing. The author hopes to accelerate progress towards practical implementation while engaging in broad communication with numerous parties.

3. Separation of Adhesive Bonds with Heat-induced Dismantlable Adhesives and Consideration of Remanufacturing

This section describes a type of dismantlable adhesive that enables the dismantling of adhesive bonds by applying heat. The material characteristics and testing of bond quality in remanufacturing are described in the same manner as the previous section.

3-1 Heat-induced Dismantlable Adhesives

Research into separation methods based on applying heat to adhesive bonds for has been carried out since the 2000s.[5)] For example, one method involves adding thermal expansion capsules inside the adhesive composition. To perform separation, heat is then applied and the capsules expand, creating pressure that causes peeling to occur (Figure 9). ThreeBond is using a similar method to develop a material that can separate adhesive bonds with one minute of heating. An important aspect in this case is that heat is a naturally-occurring trigger, meaning that there is a risk of separation outside the intended timing. Efforts are being made to reduce this risk by using separation temperatures that differ significantly from the temperatures at which components are used. This material is designed to separate when the adhesive layer reaches a temperature of approximately 300°C. Figure 10 shows the change in bond strength for two types

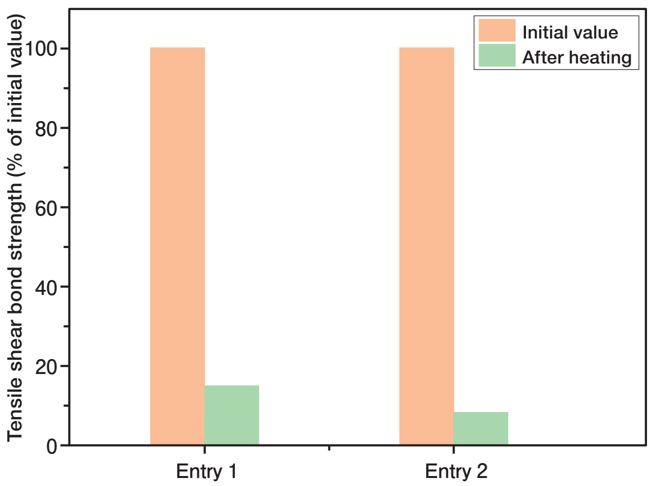
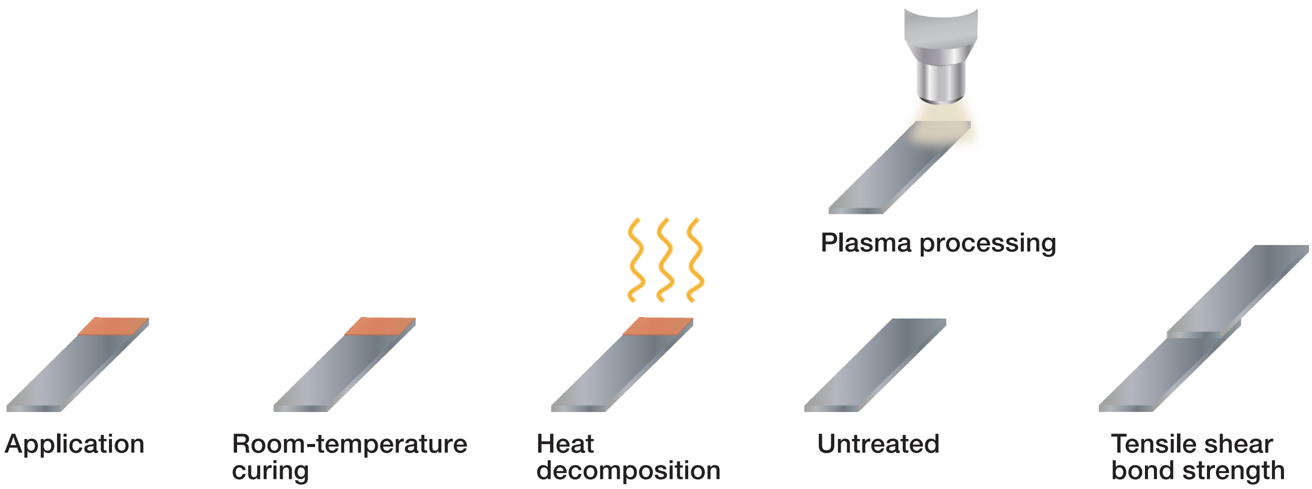


Fig. 10 Changes in Tensile Shear Bond Strength of Heat-induced Dismantlable Adhesives Before/After Heating



Adherend: A5052P aluminum; plasma processing: Nihon Plasmatreat Inc., 5 mm, 100 mm/sec; Curing conditions: RT, 1 week

Fig. 11 Experiment Workflow for Adherend Reuse Test (Heat-induced Dismantlable Adhesives)

of materials. After heating for one minute, the tensile shear bond strength decreases to about 1/5 to 1/10 its previous value. If adhesive bonds can be separated at this speed, it could be possible to create separation lines in a similar manner to production lines.

3-2 Heat-induced Dismantlable Adhesives

In a similar manner to section 2-3, this section describes the results of testing whereby heat was used to separate the adhesive bond, readhesion was attempted, and the bond strength and surface free energy before and after the process were compared.

With the heat-induced dismantlable adhesive, the experiment was performed as shown in Figure 11, and the tensile shear bond strength and surface free energy were measured at each stage. As before, these adhesive bonds did not demonstrate a reduction in tensile shear bond strength upon readhesion (Figure 12). When plasma processing is also used, the surface free energy also changes to a value similar to the initial state, which can be expected to provide even more stable adhesive bonds during readhesion.

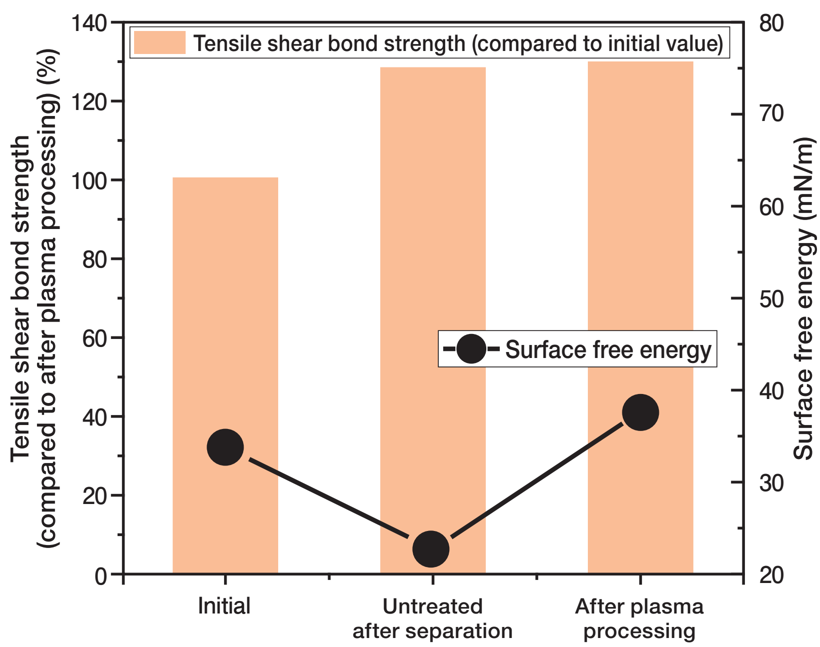


Fig. 12 Tensile Shear Bond Strength and Surface Free Energy Before and After Readhesion (Heat-induced Dismantlable Adhesives)

Closing

In this article, the author described dismantlable sealants and adhesives under development by ThreeBond, with particular focus on oxidizing agent-induced dismantlable adhesives with a non-naturally occurring stimulus, aqueous sodium hypochlorite solutions, as the separating agent and heat-induced dismantlable adhesives that separate through heating to 300°C. The article also covered ideas for future use of adhesives that considers material recycling and remanufacturing and the possibility of realizing processes that enable readhesion after adhesive bonds have been separated.

Dismantlable sealants and adhesives cannot contribute to circular economies as individual materials. They will begin to bring value when they are combined with components designed for easy disassembly. The author hopes to be able to engage in wide-ranging exchanges of opinion with those who read this article, on the road to creating materials that can contribute to society.

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