BR® 179 NON-CHROMATE BONDING PRIMER FOR AEROSPACE STRUCTURAL BONDING APPLICATIONS

Yiqiang Zhao
Solvay Composite Materials
Havre De Grace, Maryland, USA

ABSTRACT

For aerospace metal bonding applications, bonding primers are vital for enabling reliable bonding and long term corrosion protection. In order to meet the imperative market need for chromate replacement technology, technical challenges still remain for non-chromate bonding primers such as long term corrosion resistance comparable to chromated primers and improved properties at no cost to overall performance. A novel 121 °C (250 °F) curing non-chromate epoxy-based bonding primer BR® 179 has been developed by Solvay to meet these challenges and provide a path towards sustainability. Using breakthrough technology in non-chromate corrosion protection and toughening, the one-part solvent-based BR® 179 is able to provide 3000 hours long term scribe corrosion resistance of chromated-based systems, while maintaining the required level of mechanical performance. The unique resin chemistry and toughening mechanism of BR® 179 deliver improvement in low temperature (-55 °C or -67 °F) peel properties at high primer thickness (10 μm or 0.4 mil), and improved stability contributing to long shop/shelf life. This paper provides a performance data summary of BR® 179 including handling/spraying, corrosion resistance, mechanical bonding results and shop/shelf life. The compatibility of BR® 179 with various metal surface preparation methods and various epoxy-based structural adhesive systems is also highlighted.

Keywords: Non-chromate bonding primer, Corrosion resistance, Primer thickness tolerance
Corresponding author: Yiqiang Zhao

1. INTRODUCTION

1.1 Background

Bonding primers provide corrosion protection and enhanced bonding ability of metallic materials, particularly, aluminum and aluminum alloys used in the aerospace industry. Chromate containing primers offer excellent corrosion protection for aluminum alloys as well as compatibility with various metal surface preparation methods [1,2]. Typically, aluminum alloys are subject to multi-steps surface treatment before priming and bonding for durable and reliable performance. These steps include alkaline degreasing, acid etching and acid anodize process. [3] There is an urgent market request for replacing chromate-containing bonding primers with non-chromate ones due to impending or existing regulatory restrictions in different countries. However, it still remains a huge challenge for the commercial non-chromate 121 °C (250 °F) cure bonding primes to have balanced comparable performance to industrial chromated standards for aerospace metal bonding applications, i.e., solvent based BR® 127 and water based BR® 6747-1 primer.

1.2 Challenges for Non-Chromate Bonding Primers

A considerable amount of research work has been reported in the last few decades for non-chromate water based primer using non-chromate inhibitor systems.[4-11] Considering the fundamental physical chemistry dissimilarity between water and organic solvent, it is still a challenge for water based primers to have one-phase stability, sprayability and smooth film formation comparable to solvent based ones. Despite of low or zero VOC demand, there is still a market push for non-chromate solvent based primers with superior overall performance to water based ones provided the VOC elimination equipment is utilized during processing and application.

In the fabrication of aerospace structures requiring bonding a metal structure to another surface, the challenge has been to provide a primer on the metal surface that can maintain an overall balanced bonding performance meeting both bonding strength and long term corrosion protection requirements. When a primer layer is applied by spray using a spray nozzle onto the surface of a large metal section, for example, an aluminum alloy section of an aircraft having dimensions (width and/or length) of greater 1.5 meters, on the production floor, the targeted thickness for the primer layer is typically 5.1 µm (0.2 mil). Due to the limited surface area that can be covered by a single spray nozzle at one time and the variation in distance between the spray nozzle and the metal surface, it has been found that the thickness of the primer layer is not uniform across the large surface area being sprayed. Very often, there are portions or sections of the primer layer that have a thickness as thin as 2.5 µm (0.1 mil) or as thick as 7.6 µm (0.3 mil) or above. It is well known to those skilled in the bonding technology that bonding primers show unique thickness sensitivity in terms of bonding strength, particularly peel strength.

It has been found that most bonding primer products currently on the market tend to show significant peel strength drop when the primer thickness exceeds about 6.4 µm (0.25 mil). Particularly, peel strength at low temperatures such as -55 °C (-67 °F) drops even more significantly when the primer layer thickness exceeds 6.4 µm (0.25 mil). Peel strength at such low temperatures is typically required for aerospace applications. Hence, it has been a big challenge to produce bonding primers showing thickness tolerance for low-temperature peel test without negatively affecting the overall performance such as lap shear strength and handling properties. The term "thickness tolerance" in the context herein refers to the ability to withstand variation in thickness.

In terms of primer formulation chemistry, an improved primer thickness tolerance requires further toughening efficiency in the polymer networks of the cured primer layer. Moreover, any toughening material added to the primer composition to enhance such toughening efficiency should be compatible with the primer chemistry without negatively affecting the primer's overall performance. There remains a need for a method for bonding metallic structures using primer formulations that can provide high toughness in terms of peel strength at temperatures as low as -55 °C (-67 °F), and can be applied at a primer thickness of 7.6 µm (0.3 mil) or above.

Meanwhile, for the solvent based bonding primers using epoxy chemistry, the epoxy resin, curing agents and toughening additives are often dissolved in the same solvent system to form one phase solution for best film formation. However, this solution mixture may cause resin advancement concerns and limit the product shop/shelf life. Typically, the solvent based bonding primers need freezing storage with limited out time performance or shop life at ambient or high indoor temperature such as 32°C (90 °F).

1.3 Breakthrough Technology of BR® 179 Bonding Primer

In response to the above technical challenges, Solvay has developed a novel 121 °C (250 °F) curing non-chromate solvent-based bonding primer, BR® 179, to achieve breakthrough performance and long term corrosion protection comparable to the chromated primer control. Using breakthrough technology in non-chromate corrosion protection and toughening, the one-part epoxy-based BR® 179 is able to provide 3000 hours long term scribe corrosion resistance, while maintaining the required level of mechanical performance. Moreover, the unique resin chemistry and toughening mechanism of BR® 179 deliver not only significant improvement in low temperature (-55 °C or -67 °F) peel properties at increasing primer thickness as high as 10 µm or 0.4 mil, but also much improved stability contributing to long shop/shelf life. The paper summarizes the excellent overall performance of BR® 179 including handling/spraying, film properties, corrosion resistance, mechanical bonding results and shop/shelf life. Various metal surface preparation methods and epoxy-based structural adhesive systems have been employed to demonstrate the general compatibility of BR® 179 with aerospace bonding process and applications.

2. EXPERIMENTATION

2.1 Materials

BR® 179 bonding primer was formulated in Solvay's Adhesives R&I Lab. The following 121 °C (250 °F) and 177 °C (350 °F) curing adhesive products from Solvay were used in this work: FM® 94M 145 gsm(0.03 psf), FM® 94M 290 gsm(0.06 psf), FM® 73M 290 gsm(0.06 psf), FM® 300K 244 gsm(0.05 psf), FM® 377S 391 gsm(0.08 psf), FM® 309-1M 244 gsm(0.05 psf), FM® 309-1S 290 gsm(0.06 psf), FM® 289M 290 gsm(0.06 psf). All testing was done with 2024T3 Alclad and Bare aluminum alloy metal.

2.2 Metal Surface Preparation and Priming

The metal surface treatment (Ref. ASTM D 2651) consisted of MEK solvent degreasing followed by Sprex alkaline cleaning, FPL(Forest Products Laboratory) etch and Phosphoric Acid Anodizing (PAA) per ASTM 3933 or Phosphoric Sulphuric Anodizing (PSA). The primers were sprayed using Binks Model 95 or Devilbiss Model GTI-620S (HVLP) spray equipment at the desired primer thickness. Two to four box coats were used to reach primer thickness of around 3 – 7 µm. After 15 - 30 minutes air drying at 24 °C (75 °F), the primers were then cured at 121 °C (250 °F) in a preheated oven for 60 minutes.

The PSA pre-treatment of metal surface and PSA anodizing were applied with the process below:1. Solvent (Acetone) cleaning. 2. Alkaline Cleaning: Metaclean T2001, 60°C (140°F), 20 minutes, and then DI water rinse. 3. Alkaline Etching: P3-Almeco 51, 32°C (90 °F), 3 minutes, and then DI water rinse. 4. Acidic Pickling: BONDERITE C-IC SMUTGO NC AERO (former TURCO SMUTGO NC), 24 °C (75 °F), 10 minutes, and then DI water rinse.

Then PSA anodizing was done at 25°C (77°F) and 18 V for 3+20 minutes, then DI water rinse and dry in 49°C (120°F) oven for 30 minutes.

2.3 Corrosion Testing

The primed Al2024T3 Bare panels were pre-treated with FPL+PAA surface preparation and primed at 5.1 µm (0.2 mil) thickness. The primed panels were subjected to Scribe corrosion testing by salt spray exposure as per ASTM B117.

2.4 Mechanical Testing/Cure Cycles

Mechanical tests include Half Inch Lap Shear (ASTM D1002), Floating Roller Peel (ASTM D3167) and Wide Area Lap Shear (ASTM D 3165) using Al2024T3 Alclad or Bare metal with the above surface preparation.

A typical curing condition for 121 °C (250 °F) curing adhesive is: 1.7°C (3°F)/min to 121 °C (250 °F), 90 minutes at 121 °C (250 °F) under 275 kPa(40 psi) pressure. A typical curing condition for 177 °C (350 °F) curing adhesive is: 1.7°C (3 °F) / min to 177 °C (350 °F), 90 minutes at 177 °C (350 °F) under 275 kPa(40 psi) pressure.

2.5 Characterization

Differential Scanning Calorimetry (DSC) analysis was carried out on a Q20 model from TA Instruments using sample loading at 5 – 8 mg. The modulated heat ramp was applied to probe the glass transition Tg. The DSC modulated heat ramp is 30°C to 160°C at 2°C/min, ± 2 °C/40 sec. Optical photographs of tested coupons were taken with Nikon D60 Digital SLR camera. Keyence VHX-2000 3D Digital Microscope with VH-Z250R real zoom lens (x250 – x2500) was used for optical microscopic analysis in this work.

3. RESULTS

3.1 Application and Film Properties of BR® 179 Bonding Primer

Orange colored BR® 179 has 10% solid level with a homogeneous solution appearance and very small amount of powdery settlement at the bottom, as shown in Figure 1. It is noted that the one phase orange solution of BR® 179 is very stable and remains almost the same after one year storage at ambient condition. In comparison, other solvent based primers may either show increasing turbidity or discoloring at the same storage condition.

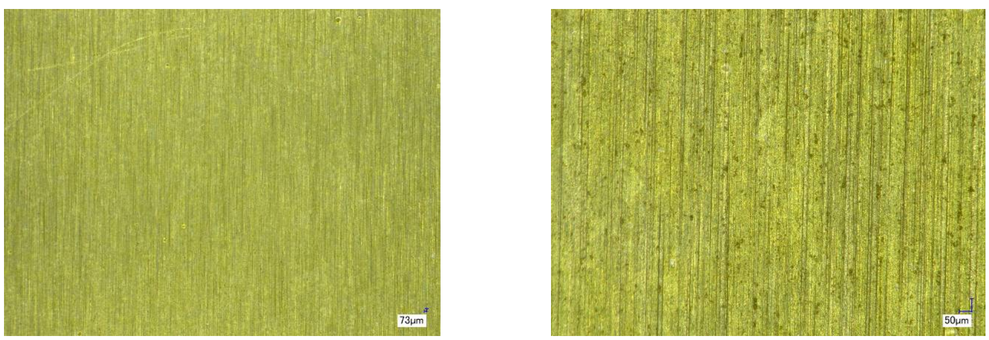
BR® 179 has comparable spraying and handling properties to BR® 127 control. It can be readily sprayed into very smooth primer film with a dark yellow (close to golden) color after air drying to easily indicate the desired primer thickness. It can be applied with a wide variety of spraying equipment including conventional spraying guns and HVLP type guns as well as hand brushing. The same spraying equipment for BR® 127 can be seamlessly utilized for spraying BR® 179. It can be readily cured at 121 °C (250 °F) in an oven for 1 hour resulting in an excellent smooth yellow film with some gloss level, as shown in Figure 1. Once cured, the film can easily pass MEK solvent wipe check, similar to BR® 127 primer film.

Figure 2 displays the optical microscopic analysis of uncured and cured BR® 179 primer film at two magnification levels to demonstrate the satisfactory smooth and homogeneous yellow film appearance using Al 2024T3 Bare(FPL + PAA). For Al 2024T3 Bare metal surface preparation with FPL + PSA and cured BR® 179 primer, the 2D and 3D microscopic analysis at x250 and x2500 magnification in Figure 3 confirms the excellent film formation of BR® 179 primer with well controlled film roughness around 1-2 μm in height profile. The well dispersed fine particulates are additives in the primer formulation as compared to pure smooth resin film. BR® 179 primer shows a typical solvent based primer film of top quality preferred by most end users.

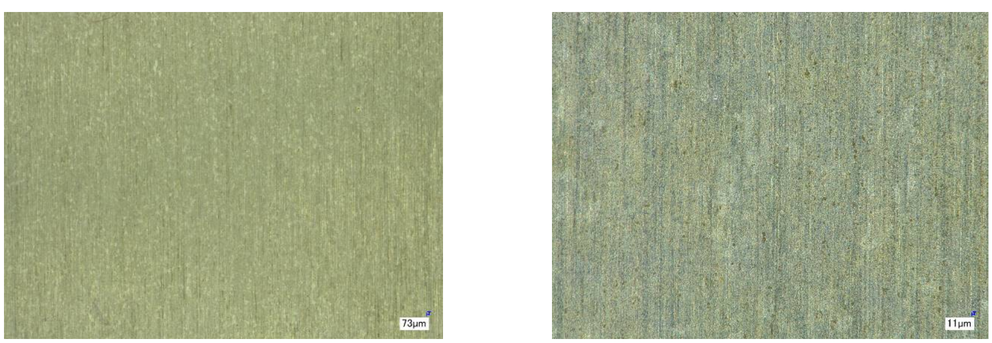
The glass transition of cured BR® 179 primer film was characterized by modulated DSC analysis in Figure 4. The obtained Tg of around 109°C is substantially higher, by more than 10°C, than most other 121 °C (250 °F) curing bonding primers. This increased Tg of cured BR® 179 primer film likely contributes to its compatibility with 177 °C (350 °F) curing bonding adhesives at high testing temperatures. Moreover, the DSC curve of oven cured BR® 179 primer film only displays a Tg transition without exothermic peak, indicating that 60 minutes curing in preset 121 °C (250 °F) oven is adequate to complete the primer curing. Due to the difficulty to completely remove the solvent during sample preparation, the effort to collect DSC profile of uncured BR® 179 primer is still ongoing to characterize the curing kinetics.

(a) (b)

Figure 1. (a) BR® 179 primer appearance; (b) Sprayed and cured BR® 179 primer film at 5.1 µm (0.2 mil) thickness using Al 2024T3 Bare(FPL + PAA).

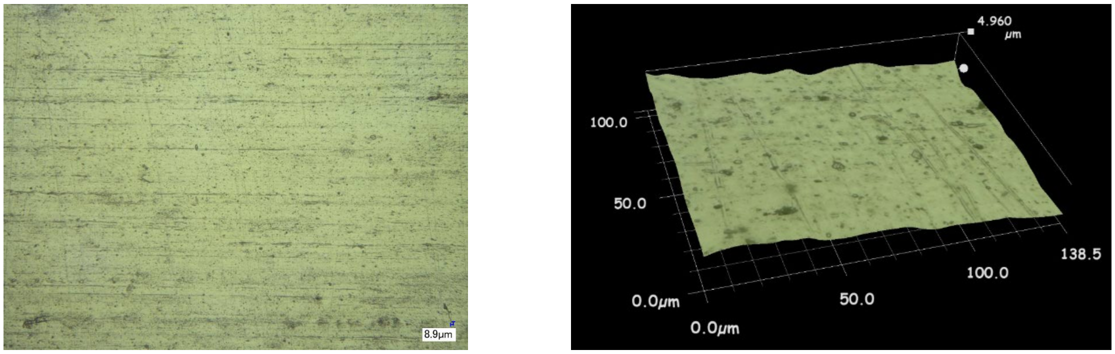


Uncured x 30 Uncured x 200



Cured x 30 Cured x 200

Figure 2. Surface micrograph of smooth and homogeneous BR® 179 primer film at 5.1 µm (0.2 mil) thickness before and after curing at two magnifications using Al 2024T3 Bare(FPL + PAA).



(a) x250 (b) 3D micrograph x250

Figure 3. Surface micrograph of smooth and homogeneous BR® 179 primer film at 5.1 µm (0.2 mil) thickness after curing using Al 2024T3 Bare(FPL + PSA).

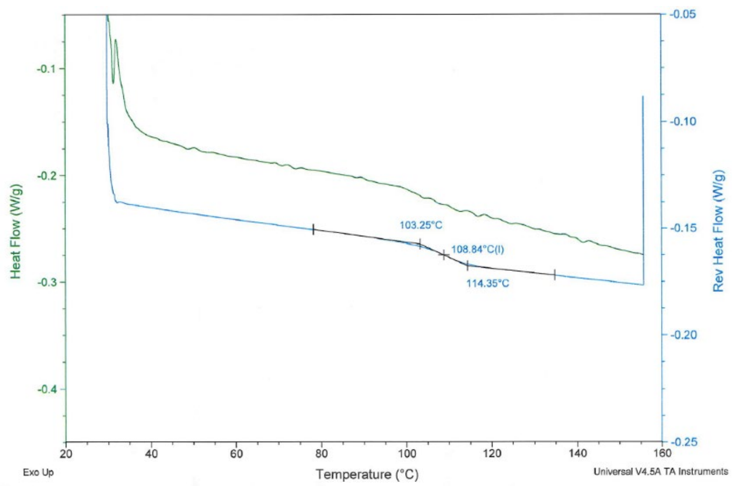
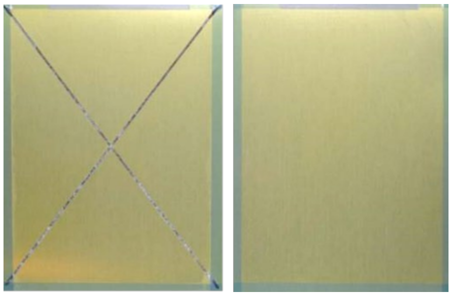


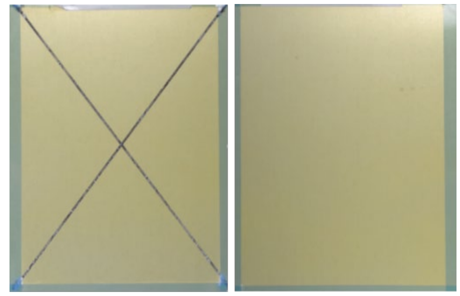
Figure 4. DSC data of oven-cured film of BR® 179 primer.

3.2 Scribe Corrosion Test

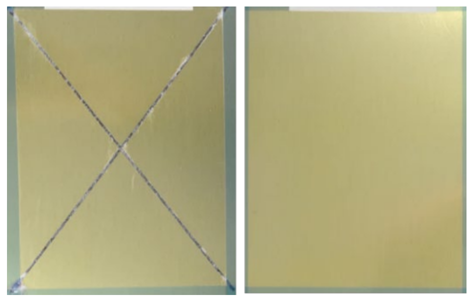
The corrosion performance of BR® 179 bonding primer has been evaluated by Scribe test under salt spray exposure as per ASTM B117 using one scribed panel and one un-scribed panel of 10 cm x 15 cm. Figure 5 shows 1000 hours, 2000 hours and 3000 hours salt spray exposure results, respectively, at 5.1 µm (0.2 mil) primer thickness for Al2024T3 Bare with FPL + PAA surface preparation. These excellent Scribe results prove the outstanding corrosion resistance of BR® 179 primer, comparable to chromated primer control. No corrosion signs are present for 1000 and 2000 hours with possible couple of undiscernible undercut dots along the scribe line in Figure 5. Only limited undercutting along the Scribe lines and very clean field surface are observed for extremely long salt spray exposure of 3000 hours, exceeding all major aerospace product specifications. The optical microscopic analysis was also performed to characterize the field area of the Scribe panel after 3000 hours salt spray, showing no field pitting or discoloring spots on the primer film with almost the same appearance as unexposed fresh primer panels (Ref. Figure 2). This clearly confirms the satisfactory anti-corrosion barrier layer of BR® 179 primer due to its excellent film formation and capable non-chromate inhibiting system. It is noted that the above Scribe test results of BR® 179 primer have already been repeated more than four times to verify the excellent long term corrosion resistance of BR® 179 primer.



1000 hours



2000 hours



3000 hours

Figure 5. Scribe corrosion data after 1000, 2000 and 3000 hours salt spray exposure for BR® 179 primer at 5.1 µm (0.2 mil) primer thickness using Al2024T3 Bare (FPL+PAA).

3.3 Mechanical Bonding Performance

BR® 179 bonding primer applies breakthrough toughening technology and unique resin chemistry to achieve a synergistic outcome in significantly improved mechanical bonding performance and stability while maintaining corrosion protection comparable to chromated primer control. The following bonding data demonstrate breakthrough primer thickness tolerance for low temperature peel test and great compatibility with both 121 °C (250 °F) and 177 °C (350 °F) curing adhesives.

3.3.1 121 °C (250 °F) Curing Adhesives

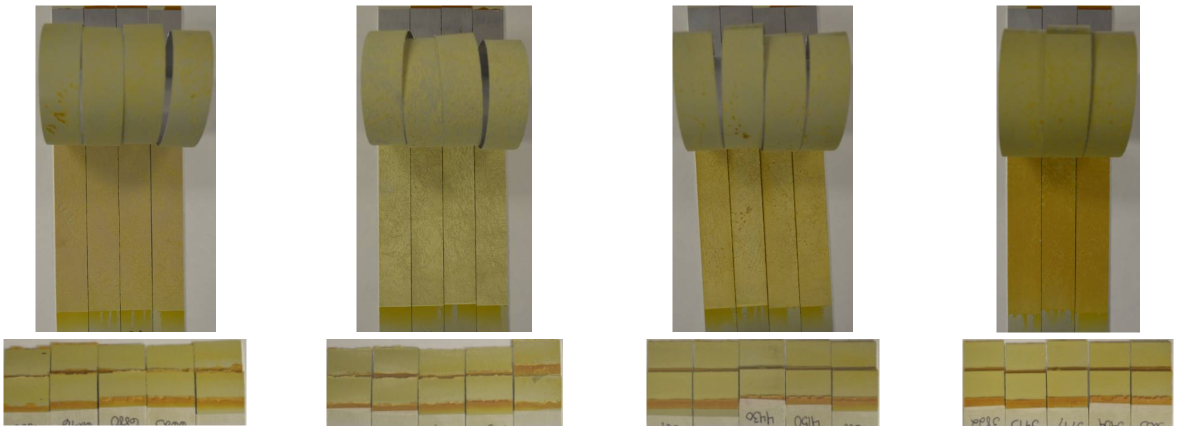
The baseline bonding test results including half inch lap shear and floating roller peel of BR® 179 primer at typical 5.1 µm (0.2 mil) primer thickness with FM® 73M 290 gsm(0.06 psf) bonding adhesive are summarized in Table 1, showing excellent shear and peel strength at various testing temperatures. Correspondingly, the failure modes of tested coupons all show 100% cohesive failure in Figure 6, including the most challenging -55 °C (-67 °F) peel test.

Table 1. Mechanical bonding data of BR® 179 bonding primer with FM® 73M 290 gsm(0.06 psf) bonding adhesive.

Test Temperature	**Half Inch Lap Shear (MPa)**	**Floating Roller Peel (kN/m)**
-55 °C (-67°F)	45.4	11.0
24 °C (75 °F)	43.0	14.0
80 °C (176 °F)	29.7	10.3

90 °C (194 °F)	25.2	8.8

* Al2024T3 Bare metal with FPL + PAA surface preparation at 5.1 µm (0.2 mil) primer thickness.



-55 °C　　24 °C　　80 °C　　90°C

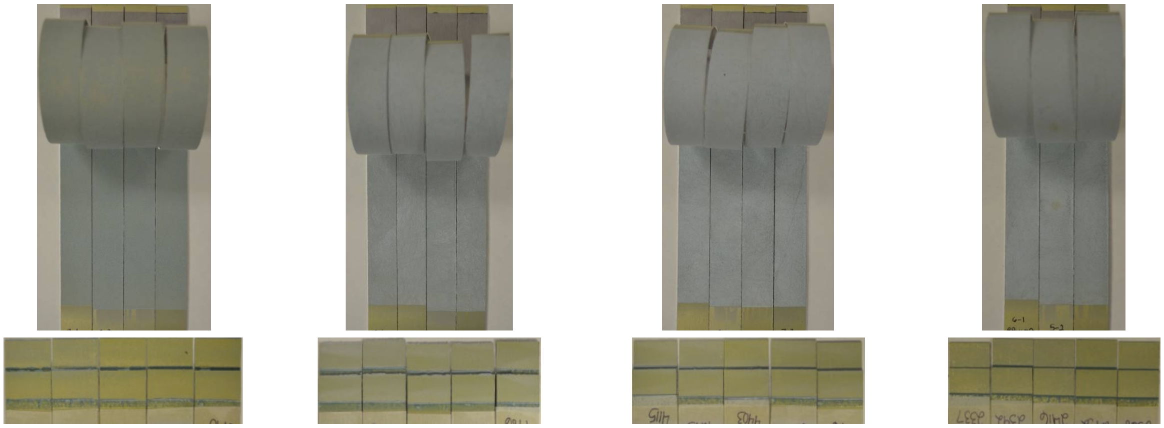
Figure 6. Failure modes of mechanical bonding data for BR® 179 primer with FM® 73M 290 gsm(0.06 psf) bonding adhesive at various testing temperatures.

The bonding results of BR® 179 primer with two grades of FM® 94M bonding adhesive are summarized in Table 2 using Al2024T3 Bare metal with FPL + PAA surface preparation at 5.1 µm (0.2 mil) primer thickness. Both adhesives show excellent lap shear and peel strength with BR® 179 at five testing temperatures. Figure 7 and 8 display the outstanding failure modes with 100% cohesive feature for lap shear and peel tested coupons.

Table 2. Mechanical bonding data of BR® 179 bonding primer with FM® 94M 145 gsm(0.03 psf) and FM® 94M 290 gsm(0.06 psf) bonding adhesive.

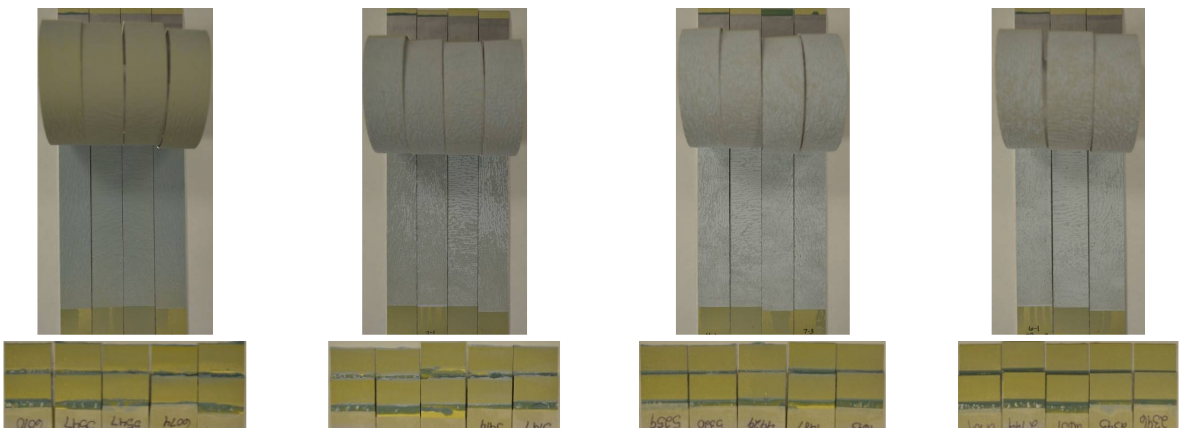
Test Temperature	**Half Inch Lap Shear (MPa)**		**Floating Roller Peel (kN/m)**	
	FM® 94M 145 gsm(0.03 psf)	FM® 94M 290 gsm(0.06 psf)	FM® 94M 145 gsm(0.03 psf)	FM® 94M 290 gsm(0.06 psf)
-55 °C (-67°F)	37.2	40.0	8.9	8.2
24 °C (75 °F)	36.0	40.0	9.1	10.7
80 °C (176 °F)	29.6	34.0	9.1	11.9
90 °C (194 °F)	27.8	31.7	8.8	11.9
121 °C (250 °F)	16.5	17.7	9.1	11.7

* Al2024T3 Bare metal with FPL + PAA surface preparation at 5.1 µm (0.2 mil) primer thickness.



-55 °C 24 °C 80 °C 121 °C

Figure 7. Failure modes of mechanical bonding data for BR® 179 bonding primer with FM® 94M 145 gsm(0.03 psf) bonding adhesive at various testing temperatures.



-55 °C 24 °C 80 °C 121 °C

Figure 8. Failure modes of mechanical bonding data for BR® 179 primer with FM® 94M 290 gsm(0.06 psf) bonding adhesive at various testing temperatures.

3.3.2 177 °C (350 °F) Curing Adhesives

The epoxy based BR® 179 primer has great compatibility with both 121 °C (250 °F) and 177 °C (350 °F) curing adhesives. The satisfactory lap shear and peel test data of BR® 179 primer with FM® 300K 244 gsm(0.05 psf) bonding adhesive are shown in Figure 9, comparable to chromated primer control. The floating roller peel data at 24 °C (75 °F) for BR® 179 primer with two more 177 °C (350 °F) curing adhesives, FM® 377S 391 gsm(0.08 psf) and FM® 309-1M 244 gsm(0.05 psf), are collected in Figure 10, also showing very good peel strength and most cohesive mode.

4.2 kN/m at -55 °C

7.2 kN/m at 24 °C

41.6 MPa at 24 °C

36.4 MPa at 80 °C

30.8 MPa at 121 °C

Figure 9. Floating roller peel(top) and half inch lap shear(bottom) data for BR® 179 primer with FM® 300K 244 gsm(0.05 psf) bonding adhesive at various testing temperatures using Al2024T3 Bare metal with FPL + PAA surface preparation at 5.1 µm (0.2 mil) primer thickness.

FM® 377S 391 gsm(0.08 psf)
6.8 kN/m at 24 °C

FM® 309-1M 244 gsm(0.05 psf)
7.7 kN/m at 24 °C

Figure 10. Floating roller peel data and failure modes at 24 °C (75 °F) for BR® 179 primer with FM® 377S 391 gsm(0.08 psf) and FM® 309-1M 244 gsm(0.05 psf) bonding adhesive using Al2024T3 Bare metal with FPL + PAA surface preparation at 5.1 µm (0.2 mil) primer thickness.

In addition, wide area lap shear was tested for BR® 179 primer and a chromated primer control with FM® 377S 391 gsm(0.08 psf) and FM® 309-1S 290 gsm(0.06 psf) bonding adhesive. The results at 24 °C (75 °F) and 177°C (350 °F) are shown in Figure 11, and BR® 179 primer either matches or exceeds the bonding strength of a chromated primer control for both FM® 377S and FM® 309-1S adhesive.

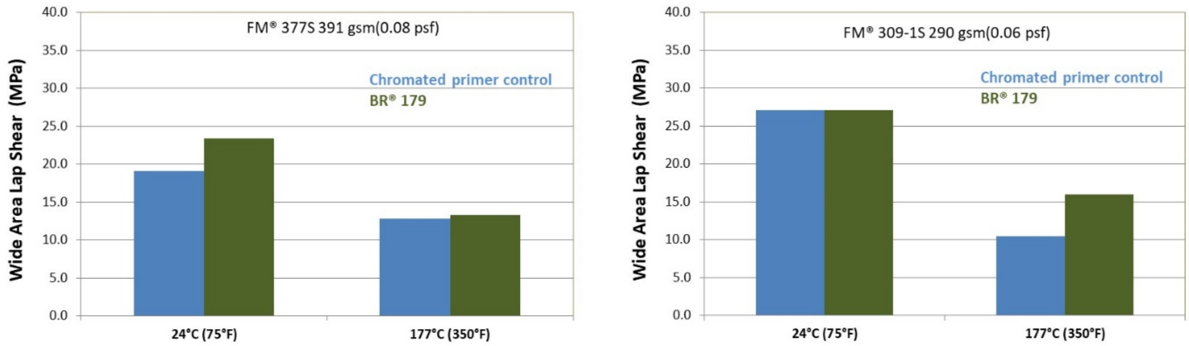


Figure 11. Wide area lap shear results for BR® 179 primer and a chromated primer control with FM® 377S 391 gsm(0.08 psf) and FM® 309-1S 290 gsm(0.06 psf) bonding adhesive using Al2024T3 Bare metal with FPL + PAA surface preparation at 5.1 µm (0.2 mil) primer thickness.

3.3.3 Primer Thickness Tolerance

It is well known that peel testing performance is sensitive to the bonding primer thickness, especially at low temperature (-55 °C or -67 °F). The -55 °C (-67 °F) peel strength at high primer thickness still remains a technical challenge for aerospace bonding applications. Most commercial primers on the market tend to show substantial -55 °C (-67 °F) peel strength drop at primer thickness higher than 6.4 µm (0.25 mil).

The floating roller peel at increasing primer thickness of BR® 179 primer with FM® 94M 145 gsm(0.03 psf) bonding adhesive was evaluated at both 24 °C (75 °F) and -55 °C (-67 °F) in Figure 12. The striking results from 2.5 µm to 10.2 µm clearly indicate very close peel strength at all thickness levels and two testing temperatures. The failure modes in Figure 13 indicate surprisingly outstanding -55 °C (-67 °F) peel robustness with almost 100% cohesive feature at high BR® 179 primer thickness, even better than low primer thickness level for FM® 94M 145 gsm(0.03 psf).

The unique new toughening technology in synergy with resin chemistry of BR® 179 primer gives rise to this breakthrough performance, which has been further confirmed by more bonding conditions with more 121 °C (250 °F) curing adhesives. Floating roller peel results at high primer thickness 7.6 µm (0.3 mil) for BR® 179 with FM® 73M 290 gsm(0.06 psf), FM® 94M 145 gsm(0.03 psf) and FM® 289M 290 gsm(0.06 psf) bonding adhesive are presented in Figure 14, using Al2024T3 Alclad with PSA pretreatment and PSA surface preparation. The obtained -55 °C (-67 °F) peel strength values are almost the same as 24 °C (75 °F) ones for all three adhesives at high primer thickness 7.6 µm (0.3 mil), showing similar cohesive dominant failure modes.

The above robust bonding results at high primer thickness unambitiously prove that BR® 179 primer delivers a breakthrough solution to the long time technical challenge in adhesive bonding applications, by enabling the outstanding primer thickness tolerance in primer processing on the production floor to ensure much improved reliability in bonding performance.

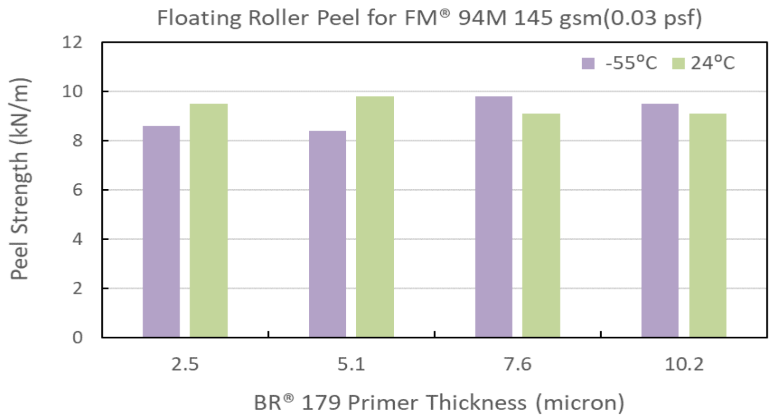


Figure 12. Floating roller peel results for BR® 179 primer and FM® 94M 145 gsm(0.03 psf) bonding adhesive at various primer thickness levels using Al2024T3 Alclad with FPL+ PSA.

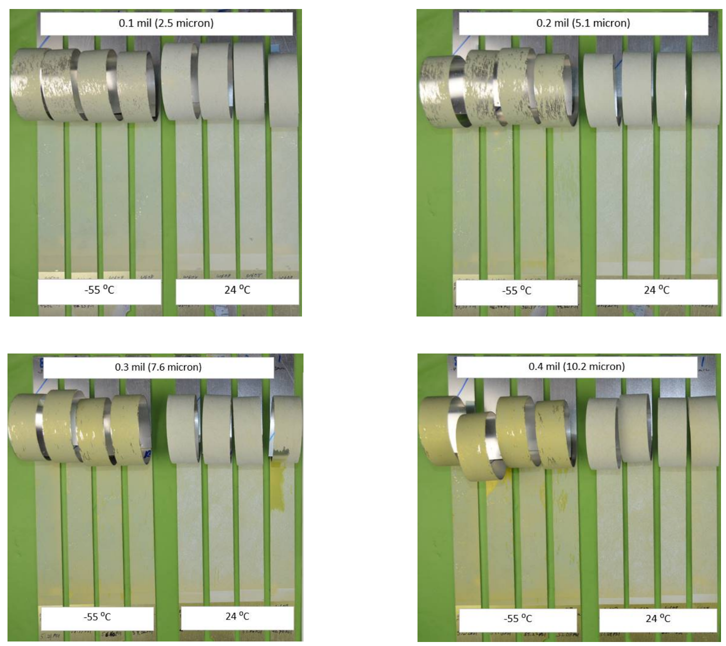


Figure 13. Floating roller peel failure modes for BR® 179 primer and FM® 94M 145 gsm(0.03 psf) bonding adhesive at various primer thickness using Al2024T3 Alclad with FPL+ PSA.

11.9 kN/m at 24 °C | 7.9 kN/m at -55 °C — FM® 73M 290 gsm

8.4 kN/m at 24 °C | 8.4 kN/m at -55 °C — FM® 94M 145 gsm

9.1 kN/m at 24 °C | 10.0 kN/m at -55 °C — FM® 289M 290 gsm

Figure 14. Floating roller peel results for BR® 179 primer at high primer thickness 7.6 µm (0.3 mil) with FM® 73M 290 gsm(0.06 psf), FM® 94M 145 gsm(0.03 psf) and FM® 289M 290 gsm(0.06 psf) bonding adhesive using Al2024T3 Alclad with PSA surface preparation.

3.4 Shop and Shelf Life

The current one-part epoxy and solvent based bonding primers on the market tend to have limited shop life at ambient or high temperature and often need freezing storage, due to the resin advancement in the presence of curing agents. In contrast, as discussed in Sec 3.1, BR® 179 has outstanding stability and can maintain the one phase orange solution appearance after even one year storage at ambient condition. The unique resin formulation chemistry and fully synergistic toughening mechanism in BR® 179 enables this much improved breakthrough out time stability and long shop/shelf life.

3.4.1 Out Time Stability

For bonding tests, it is well known that the peel performance, especially -55 °C (-67 °F) peel, is much more challenging than lap shear. Thus, the out time floating roller peel data at typical 5.1 µm (0.2 mil) primer thickness have been developed in Figure 15 and 16 to demonstrate the great breakthrough stability of BR® 179 at ambient condition, ca. 24 °C (75 °F), and hot environment, ca. 32 °C (90 °F), using two typical 121 °C (250 °F) curing adhesives, FM® 94M 145 gsm(0.03 psf) and FM® 73M 290 gsm(0.06 psf). The out time peel results at three testing temperatures in Figure 15 indicate the same peel strength even after 180 days out time on the bench at ambient condition. For elevated out time condition at 32 °C (90 °F), BR® 179 can also impressively deliver very solid peel performance at -55 °C (-67 °F), 24 °C (75 °F) and 80 °C (176 °F) even after 180 days in Figure 16. In contrast, most current one-part epoxy and solvent based bonding primers tend to show less than 15 or 30 days shop life at 32 °C (90 °F). This much improved out time stability allows BR® 179 to show very long shop life at ambient and elevated temperature, leading to the outstanding flexibility and time/cost savings with primer handling and application as well as shipping/transportation.

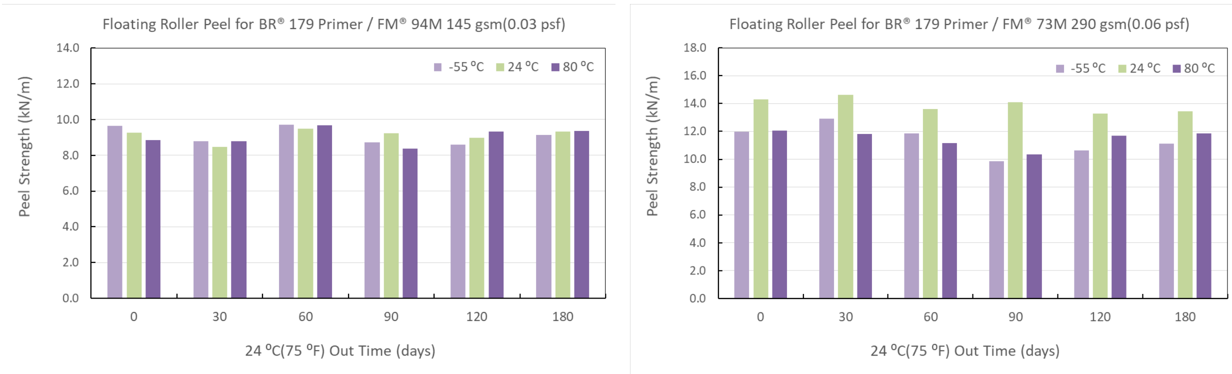


Figure 15. Floating Roller Peel data for BR® 179 out time performance at 24 °C (75 °F) and ambient humidity (~ 58 % RH) using Al2024T3 Bare with FPL + PAA surface preparation and two bonding adhesives.

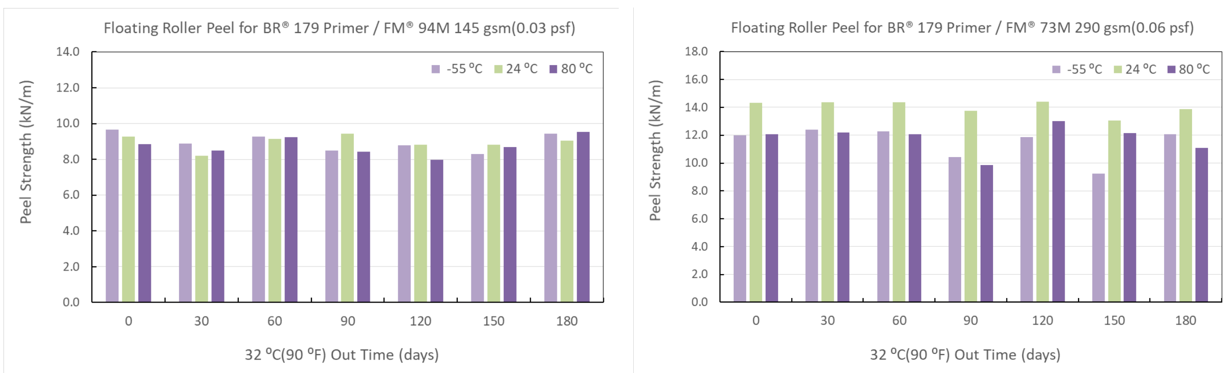
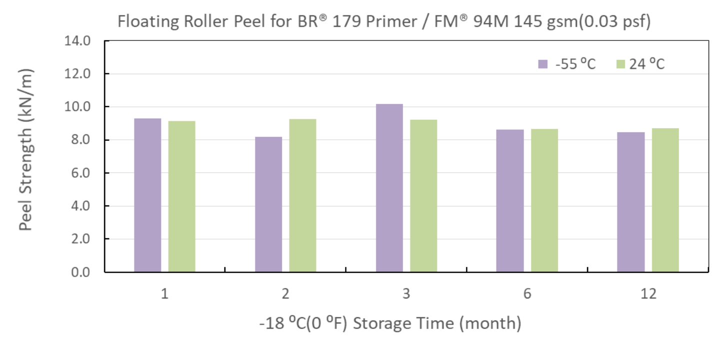


Figure 16. Floating Roller Peel data for BR® 179 out time performance at 32 °C (90 °F) using Al2024T3 Bare with FPL + PAA surface preparation and two bonding adhesives.

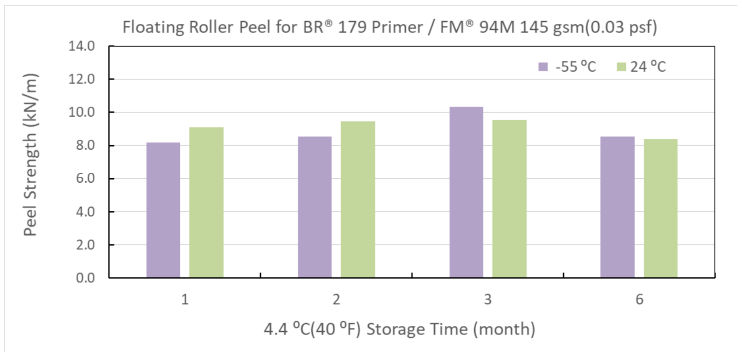
3.4.2 Shelf Life

Consistent with the breakthrough shop life above, the superior stability of BR® 179 gives rise to its excellent long shelf life as well. The 12 months shelf life peel data of BR® 179 at 5.1 µm (0.2 mil) primer thickness have been collected in Figure 17(a) for typical freezer storage at -18 °C (0 °F) using FM® 94M 145 gsm(0.03 psf) bonding adhesive and Al2024T3 Alclad with FPL+ PSA surface preparation. The solid peel strength results at -55 °C (-67 °F) and 24 °C (75 °F) after one year storage at -18 °C (0 °F) evidently prove that BR® 179 can readily pass typical one year shelf life requirement using freezer storage at -18 °C (0 °F). Moreover, the shelf life at -18 °C (0 °F) may likely to be further extended providing further data development.

Furthermore, the possibility of BR® 179 storage at refrigerated condition, ca. 4.4 °C (40 °F), has been evaluated with the collected peel data shown in Figure 17(b). The bonding results at -55 °C (-67 °F) and 24 °C (75 °F) testing temperature after 6 months storage at 4.4 °C (40 °F) remain almost the same peel strength as the relatively fresh primer, suggesting at least 6 months or likely even longer shelf life at refrigerated storage at 4.4 °C (40 °F). This means BR® 179 can also be stored in a refrigerator during frequent use period to save handling time and production cost as compared with freezing storage.



(a) Freezer storage at -18 °C (0 °F)



(b) Refrigerated storage at 4.4 °C (40 °F)

Figure 17. Floating Roller Peel data for BR® 179 shelf life performance using (a) freezer storage at -18 °C (0 °F) and (b) refrigerated storage at 4.4 °C (40 °F). FM® 94M 145 gsm(0.03 psf) bonding adhesive was employed using Al2024T3 Alclad with FPL+ PSA surface preparation.

4. CONCLUSIONS

The non-chromate solvent-based BR® 179 bonding primer is a novel one-part 121 °C (250 °F) curing bonding primer developed by Solvay to show breakthrough performance using a unique epoxy based resin formulation chemistry and innovative toughening technology. The unique synergy between resin chemistry and toughening mechanism is a key to this breakthrough technology. BR® 179 with 10% solids has excellent handling/application properties with remarkable out time stability and very long shop/shelf life based on solid bonding performance data, superior to current one-part epoxy and solvent based bonding primers in the market. It can be readily sprayed with conventional or HVLP type of spray guns, or applied by hand brushing, resulting in great film formation and highly smooth and homogeneous yellow film before and after curing through 3D optical microscopic analysis. BR® 179 primer has excellent corrosion resistance with 3000 hours Scribe test result comparable to chromated primer control. The abundant developed mechanical data of BR® 179 primer demonstrate the robust bonding performance and great compatibility with a wide variety of 121 °C (250 °F) and 177 °C (350 °F) curing adhesives as well as various metal surface preparations. Particularly, the unique toughening mechanism of BR® 179 realizes the robust improvement in low temperature (-55 °C or -67 °F) peel properties at increasing primer thickness as high as 10 µm or 0.4 mil. BR® 179 primer delivers a breakthrough solution to the long time technical challenge in adhesive bonding applications, by enabling the outstanding primer thickness tolerance in primer processing on the production floor to ensure much improved reliability in bonding performance.

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