

# **EPOXY RESIN AND RELEASE AGENTS PART I: INFLUENCE OF EXTERNAL AND INTERNAL RELEASE AGENTS ON THE ADHESIVE PROPERTIES OF EPOXY RESIN**

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*A wide range of epoxy resins are molded to produce composite components for various applications. Release agents are generally used to facilitate the removal of the component from the mold. The work presented in this paper shows differences between release agents and resin during a RTM process. Adhesive properties of release agents in interaction with the resin are analyzed, with respect to pressure and temperature. It was found that the reaction of epoxy resin results in indifferent breakaway forces from mould surfaces depending on used agents.*

*Keywords: Epoxy resin, internal and external release agent, RTM (Resin transfer molding) process*

## **INTRODUCTION**

Epoxy resin is one of the most important polymers, with applications in such fields as structural adhesives, surface coatings, composite materials and electronic devices and so on [03]. The interest in epoxy resins is related to their attractive characteristics, including small shrinkage during curing, good adhesion to substrates and electrical and mechanical resistance [13]. Moreover, epoxy resins have good thermal, dimensional, as well as electrical stability and, chemical resistance. [03,05]. Epoxy composites market is very fast evolving particularly for the high-performance composites. This is characteristic in the automotive industry for short cycle time as challenge for mass production [16]. The resin transfer molding (RTM) process is one of the widely used manufacturing processes of composite structures in the industry of transport (e.g. automotive) and aircraft [16]. Lately that process has more attention in industry of many advantages [02]. The material cost with RTM is much lower than with prepreg technology. It creates a compromise in comparison of manual lamination, autoclave molding and RTM process in terms of requirements, productivity and quality [10]. The typical RTM process cycle consists of four phases:

- The 1<sup>st</sup> phase is preform manufacturing which produces the fiber reinforcement in

the shape of the finished part.

- The 2<sup>nd</sup> phase is mold filling during which resin is injected into the cavity of a mold containing the preplaced preform.
- The 3<sup>rd</sup> phase is resin curing which may start during or after the second phase.
- The 4<sup>th</sup> phase is demolding which occurs after the curing reaction comes to completion and the part solidifies [01].

One of the important points of resin composites is to select release agents properly according to interactions between resin and both mold surfaces and fibers. Most often these components are either mixtures of esters, waxes, zinc and calcium stearates or fluorocarbon and silicon complexes [09]. Polymeric components are molded to particular shapes and release agents are used to facilitate removal of the component from the mold [15]. The agent creates a very low surface energy coating on a substrate, which allows the shaped material to be released quickly and easily. Applied release agent causes elimination or reduction of adhesion on the surface of the mold, which is often difficult to achieve [14,17]. A well preparation and precise application of these products during manufacturing process is very important. It takes time to prepare a clean surface of the mold faultlessly and the right amount of agent has to be applied. To be effective, the surface must be cleaned thoroughly to remove all traces of oils,

,waxes or other impurities [04]. Release agents can be divided into two groups: external- products which are applied on the mold surface; and internal- which are combined with the resin system. Application of external release agents is critical in ensuring their optimal performance. It can be applied by hand or using spray equipment. Traditional agents with problems that have already been resolved like safety concerns due to environmental and health issues have led to new alternative water-based products. The major disadvantage of the water-based release agents is a long time of evaporation and it is recommended to use them with heated molds. The recent alternative is to use a semi- permanent release agent [12], which are reactive resin solutions designed to cross- link and cure on the mold, providing inert release films[11,12].

Internal release agents are combined with the resin prior to molding. The agent is dissolved in the mixed resin. The time to apply an external agent on the surface compared to an internal agent is eliminated from the production process. One main advantage of this type of release agents is robustness of process for high volume manufacturing[12].

This paper presents the differences between the impact of the external and internal release

agents on the epoxy resins of the molding polymer. In practice, there are no suitable tools which can effectively classify effects of release agents. Moreover, the influence of release agents on adhesion between mold surface and epoxy resin is investigated. The aim of this work was to identify suitable release agents for epoxy resins.

## EXPERIMENTAL

### Materials used

A standard epoxy resin of bisphenol- A (epichlorhydrin) type, suitable for construction and industry, with an average molecular weight of 700, supplied by Sika®, was used. The hardener 3-aminomethyl- 3, 5, 5- trimethylcyclohexylamine, 2- piperazin- 1- ylethylamine, was modified by weight in a ratio of 100 to 24.

All release agents used in this paper are characterized in Table 1.

### Preparation

All samples were prepared by mixing a formulation of resin and hardener.

External release agent was applied to mold surface at room temperature. The mold was covered by triple coating layer of an external agent. Before

Table 1: Characterisation of release agents.

Producer/ Provider	Products
External release agent	
Axel Plastics Research Laboratories Ins (www.axelplastics.com)	XTEND 19RBU®- semi- permanent releases.
Chem- Trend (www.chemtrend.com)	Mold Sealer S-31® and Treil Part 310®-semi- permanent release agents.
Hacotech (www.hacotech.com)	Hacowax T1®- external release agent.
Henkel (www.henkelna.com)	Frekote 55 NC®, Frekote 700 NC® and Frekote 770®- semi- permanent release agents.
KVS Eckert & Woelk (www.kvsewo.de)	EWOMold 3721® and VP 094491®- external release agents.
Muench Chemie International (www.muench-chemie.com)	Mikon 705 MC®, Mikon 34+® and Mikon W 38+®- semi- permanent release agents.
Zyvax Inc (www.zyvax.com)	Watershield® and Enviroshield ®- solvent- free semi- permanent release agents.
Internal release agent	
WELA Handelsgesellschaft (www.wela-handelsgesellschaft.de)	Additive G- 161® and Additive G- 161®- internal release agents.
E. and p. Wuertz & Co KG (www.epwuertz.de)	PAT®657/ BW® and PAT®657/ BW- internal release agents.

applying an external release agent the surface has been thoroughly cleaned (Figure 1a). The sealer sealed effectively all micro porosity on the tool surface (Figure 1b). The release agent was applied as a smooth wet film over an area in accordance to the specific instructions (Figure 1c). The internal release agents were mixed with epoxy resin and hardener. The added amount of agents (1vol%, 2vol%, 3vol% and 5vol%) is related to the final mixture. For every test a 2 g sample was prepared. The samples were exposed at temperature of 80°C and pressure of 1MPa and were cured for 20 min. To facilitate the cleaning of the mold's surface Surface Cleaner® and Mold Cleaner #3® were used to clean surfaces after every test. The epoxy resin was applied on the

molds surface (diameter 90 mm). The used material was grinded steel (Figure 2b).

### Testing method

To test the external and internal release agent special geometry was designed and integrated in the testing machine (Figure 2a). Base design is similar to the approach of University of Bayreuth [09].

Tests were carried out on an universal testing machine model Instron 5567 under controlled atmosphere conditions (23°C ±2 K) and a cross-head speed of 1 mm/min<sup>-1</sup>. Inside the mold, there were two heating elements (bottom and top) and one sensor PT100 in each plate to control temperature (Figure 2b).

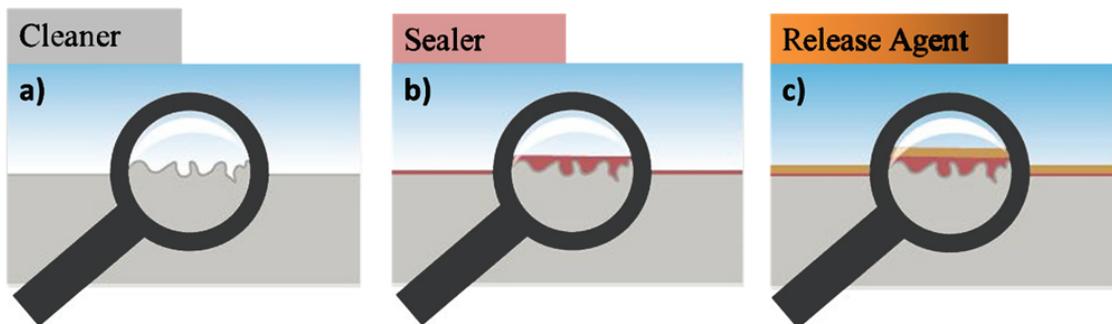


Figure 1: Applying the release agent on the surface: a) cleaner, b) sealer and c) release agent [16].

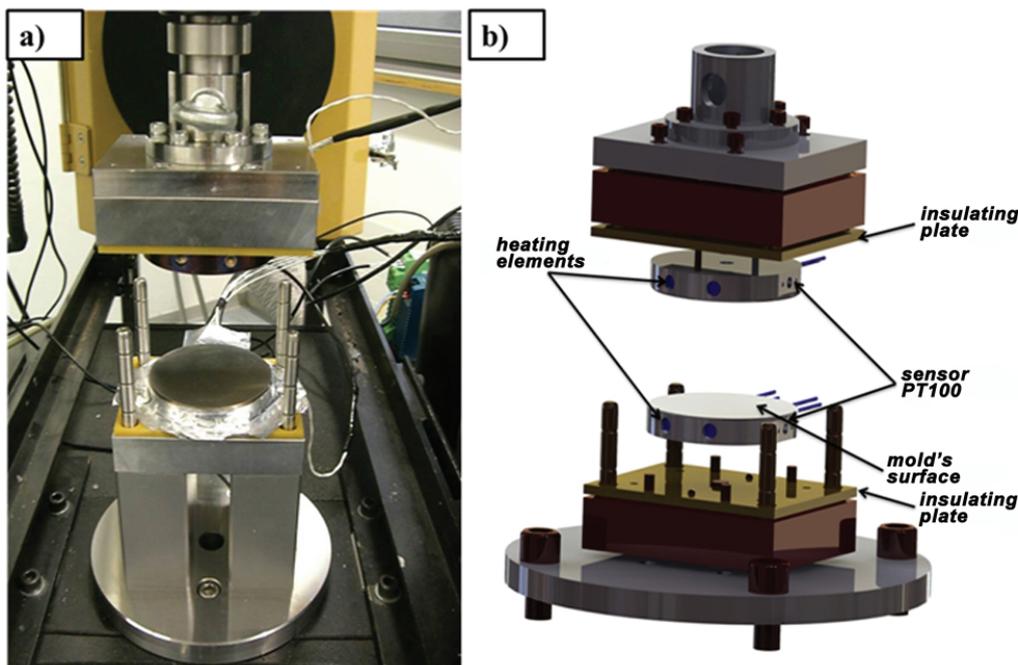


Figure 2: An instruments a) Instron 5567 used for measurement, b) image of test stand in SolidWorks.

All components were connected with the heating devices as shown in (Figure 3).



Figure 3: The heating device for heating elements and sensors

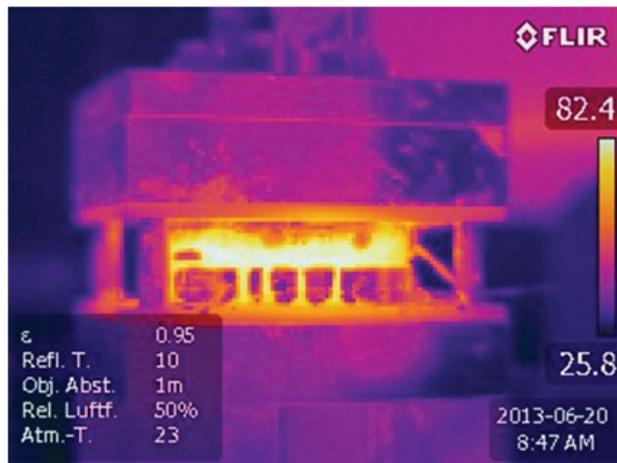


Figure 4: Thermographic image of the heating surfaces

The temperature accuracy between the mold surfaces was  $80^{\circ}\text{C} \pm 2\text{ K}$  (Figure 4). The epoxy resin was applied, after mixing, to the bottom part. Defined distance between two mold surfaces was 0.5 - 0.7 mm. After completing, the epoxy resin has to be separated from the mold surface.

### RESULTS AND DISCUSSION

The analysis allowed determination of influence both of the external and internal release agents on the behavior of a resin system in automated industrial processing. The differences in demolding forces emphasize that chemistry (which is usually not ordinarily known), must be considered for each system.

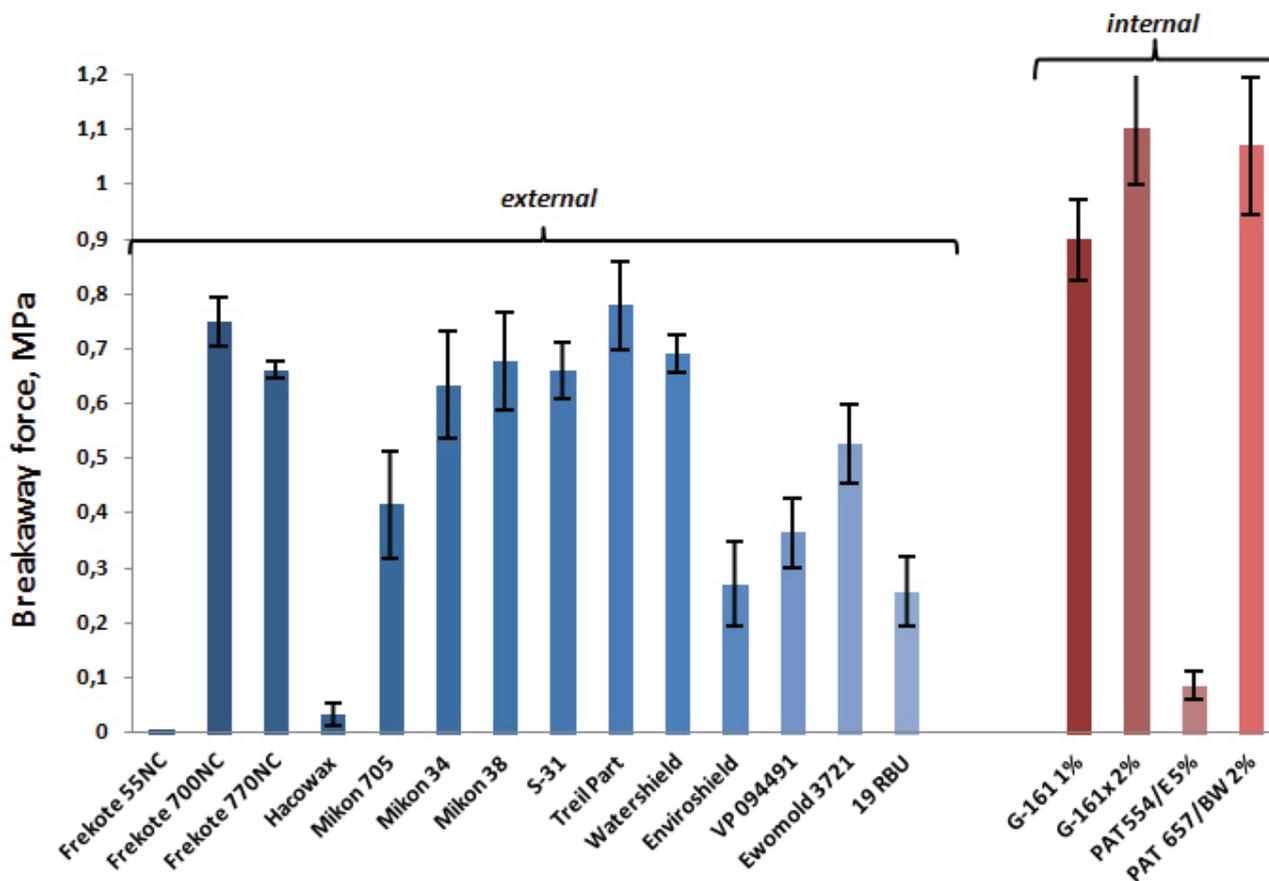


Figure 5: Breakaway force compared to different external and internal release agents

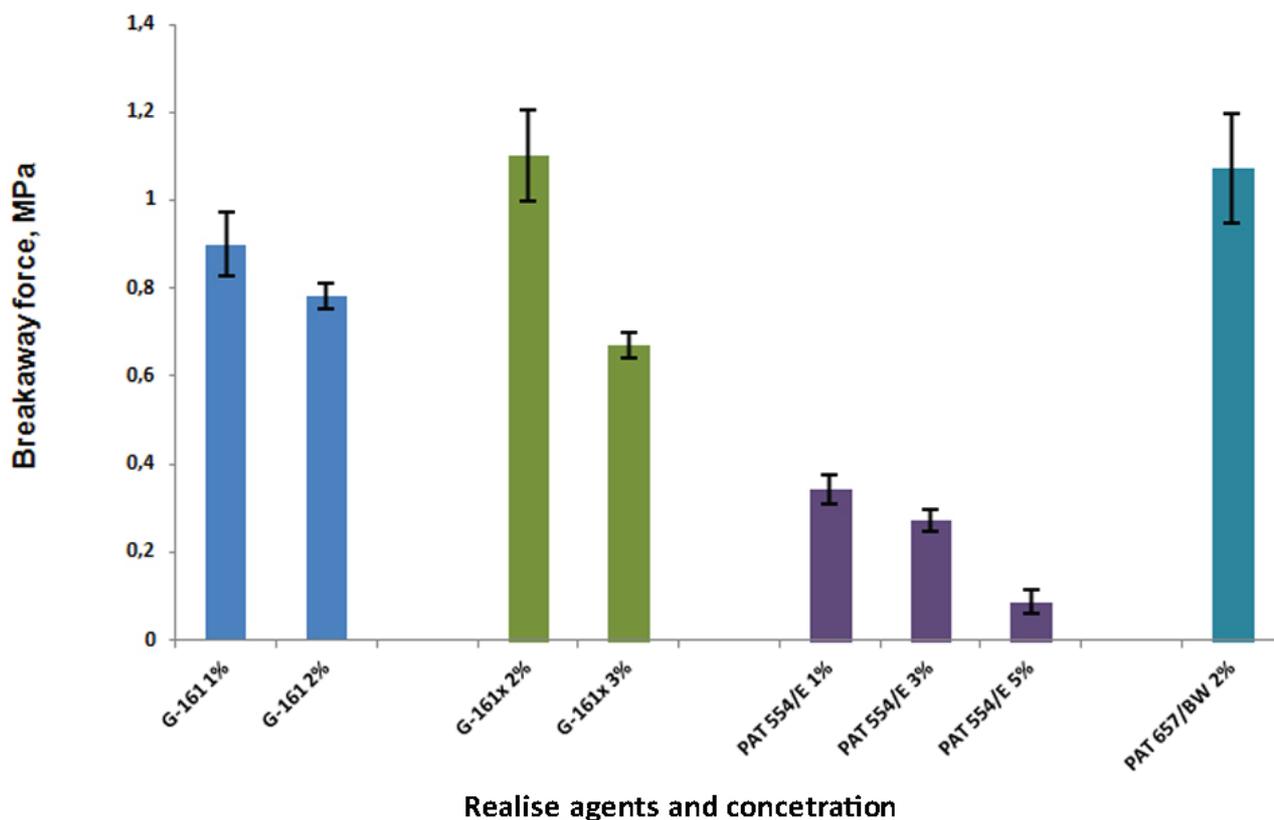


Figure 6: Breakaway force with different concentration of % internal release agents

However, after the same time, the external release agent was not on the mold surface and the application process took time. Due to the impossibility of performing measurements, the fact that all applied coats were different, needs to be taken into consideration. The assessment shows that the presented method does not allow obtaining reproducible thickness of coatings.

It was proved that only one internal release agent PAT 554E with 5% of concentration is comparable to external release agents. It can be observed that Frekote 55 NC of 0,0045 MPa shows the low surface energy on a surface compared to Frekote 700 NC of 0,74 MPa.

The below-presented graph demonstrates that not every release agent is suitable for this epoxy resin.

Figure 5 presents different amounts 1%, 2%, 3% and 5% of internal release agents. The higher % concentration is determined by producer. There is a clearly defined pattern to the graph, and this can be taken to mean that with higher amount of % is the lowest surface energy. Nevertheless, Fig. 6 shows that minimum and maximum concentration of release agent has an influence on the results. Moreover, the release agent PAT 554 E of 5% presents the lowest energy on surface

under force of 0,08 MPa. The release agent PAT 554 E (1%, 3%, 5%) shows the best result compared to G-161, G-161x and PAT 657 BW.

### CONCLUSION

This paper presents the influence of the external and internal release agents on the breakaway force of the bisphenol-A (epichlorhydrin) epoxy resin. The results clearly show that it is impossible to find an universal release agent for the epoxy resin. The difference between two release agents shows that only one of the external and internal release agents can create a very low surface energy on a substrate. It is shown that concentration of % internal release agent has a considerable impact on the results. With the rise of content of the internal release agent then decreases the value of force acting on the surface. However, repetition of the same % content of the agent does not correspond to the comparable result with the other agent. It depends on the chemical composition of substance, which is usually not given by manufacturer and on the chemical composition of resin with which it is used. Designation of external and internal release agent into existing reactive epoxy resin is an important step towards process automation.

Future work will be focused on the testing of different adhesive agents using a wide range of surfaces, higher temperatures and comparison of the similar epoxy resin to the one presented in this paper.

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### REFERENCES

- 1) Advani S. G., Brusche M. V., Parnas R., Flow and rheology in Polymeric Composites Manufacturing , (Ed. Advani S. G), Elsevier, Amsterdam 1994, pp. 466- 479
- 2) Archim V., Ruiz E., Int J Master Form 2009,3 (S2), pp. 1277-1286
- 3) Coscaval C. N., Rosu D., Mititelu- Mija A., Rosu L., Polimery 2006, 51, pp. 199-205
- 4) Goss B., Reinforced Plastics 2004, 8, pp. 24-26
- 5) Hodkin J. H., Simon G. P., Varley R. J., Polym. Adv. Technol. 1998, 9, pp. 3-10
- 6) [http://www.lange-ritter.de/fileadmin/user\\_upload/Downloads/Produkte/Diverses/L\\_R\\_Katalog\\_2013\\_Diverses\\_Formentrennmitel.pdf](http://www.lange-ritter.de/fileadmin/user_upload/Downloads/Produkte/Diverses/L_R_Katalog_2013_Diverses_Formentrennmitel.pdf) (access 2013)
- 7) Kang M. K., Jung J. J., Lee W. I., Composites Part A 2000, 31(5), pp. 407- 422
- 8) Karbhari M. V., Journal of material science letters 1998, 17, pp.2061-2062
- 9) Krämer J., Hähnel M., Fabris W., Altstädt V., „Classification of internal release agents-Evaluation of test facility and the effect on the neat epoxy resin properties”, Thermosets, Berlin, Germany, 22 September, 2011
- 10) Naik K. N., Sirish A., Inani A., Progress in Aerospace Sciences 2014, 65, pp. 22-40
- 11) Plastic, Additives and Compounding, Volume 1, Issue 1, April- May 1999
- 12) Reinforced Plastics, Volume 52, Issue 7, July- August 2008
- 13) Rosu L., Ciobanu C., Rosu D., Polimery 2008, 53, pp. 644-648
- 14) Scyidow F. T., Mansoori Y., TaghiNezhad J., Industrial Lubrication and Tribology 2007, pp.236-241
- 15) Shield A. J, Hpburn D. M., Kemp J., Cooper J. M., Polymer Degradation and Stability 2000, 70, pp.253- 258
- 16) Workshop, Leichtbaufertigung mit Faserbundwerkstoffen, Ditzingen, Germany, 20 November, 2012
- 17) Würtz J., „Leistungs- und Qualitätssteigerung in der Serienproduktion durch interne Trennmittel“, 17 Nationales Symposium SAMPE Deutschland e. V- Hochleistung und Großserie, Aacheln, Germany, 16-17 February, 2011

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