

JOINING OF PLASTICS BY ADHESIVE BONDING – WHAT NEEDS TO BE CONSIDERED

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INTRODUCTION

We live in a world where plastics are omnipresent and can be found in almost every product of everyday life. There are many reasons for this - for example, their simple processing and low density. They are also inexpensive and often require less energy than alternative materials in their manufacture and processing. In a large number of cases, however, a product does not consist of just one (plastic) part, but is composed of components made from different materials. In order to form the ready part components need to be joined together. Modern bonding technology is the predestined process for joining different materials but as for every joining technique, the necessary technical characteristics must be fulfilled and at the same time high productivity, quality and reliability must be ensured. From the multiple aspects to be considered when designing a bonding process (Fig. 1) this article will focus on the interaction between the plastic parts to be joined and the adhesive required to form a reliable bond.

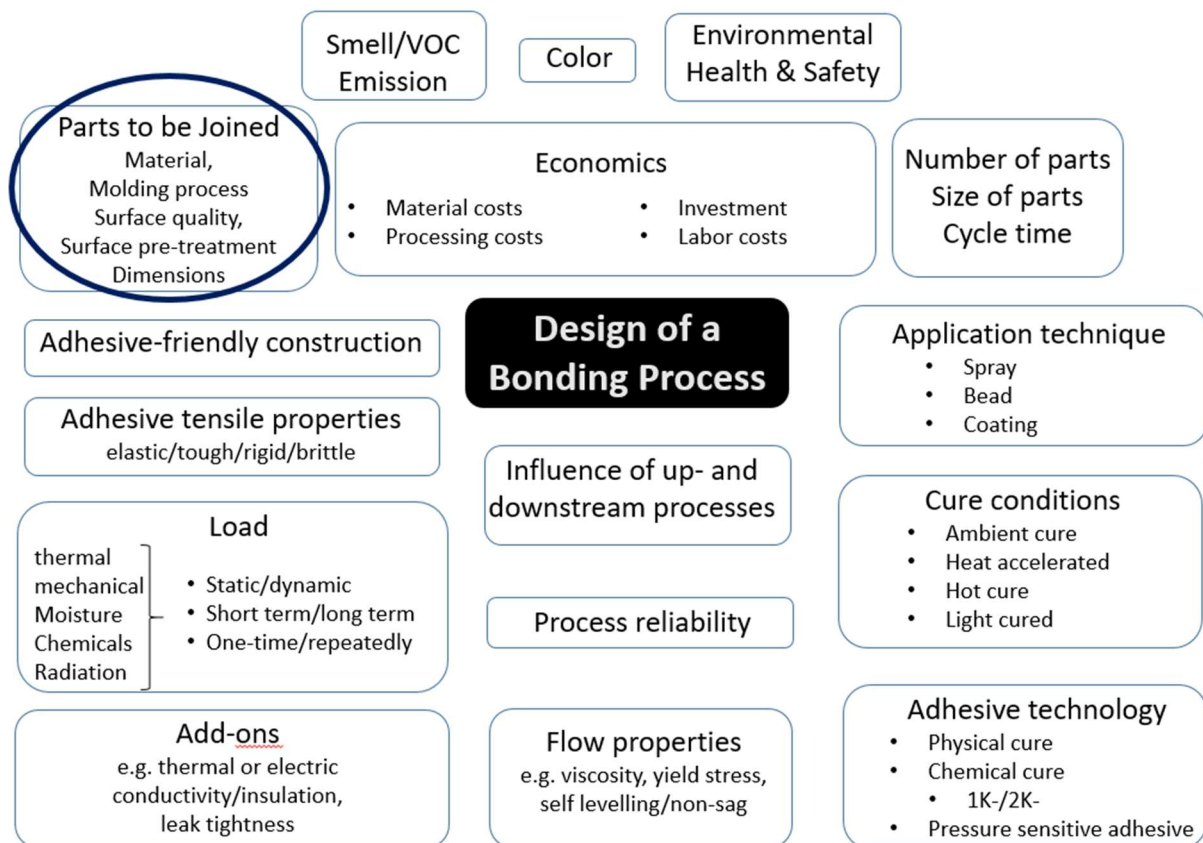


Fig. 1: Overview of the various parameters to be taken into account when designing a bonding process

WHICH EFFECTS INFLUENCE THE BONDABILITY OF PLASTICS?

The large number of plastics available on the market which are in use for various applications in different industries do not allow any general statement regarding bondability of plastics. Without question bondability depends on the type of polymer, but there are other factors that must be taken into

account as well. The formation of adhesive forces is a question of surface properties of the part which needs to be bonded. The adhesive forces, which ultimately determine whether an adhesive provides sufficient adhesion to a plastic surface, show only a very short range of less than 1 nm. These adhesion forces are based on the interaction of functional groups present on the part's surface with corresponding groups present in the adhesive. This can be polar or polarizable molecule groups (Fig. 2), groups being capable to form hydrogen bridge bonds or to allow for the so called van der Waals forces. Having the short range of such forces in mind it is obvious that adhesive and part surface has to come in close contact to allow interaction. A prerequisite is a good wetting of the surface by the adhesive, which requires that the surface tension of the part surface needs to be higher than the one of the adhesive in a liquid state. In addition it is required that the functional groups on the part surface can interact with those present in the adhesive – surface and adhesive have to match each other.

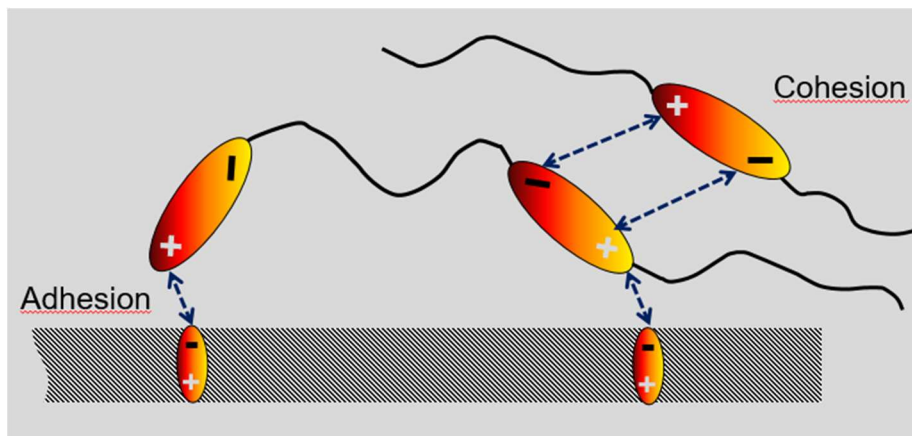


Fig. 2: Dipole-dipole-interaction resulting in adhesion forces between part surface and adhesive and cohesion within the adhesive

As already mentioned the type of base polymer plays an important role but plastics as synthetically produced materials are typically formulated for different reasons with a wide variety of additives which can be pigments, fillers or fibers, but also internal release agents, plasticizers, antistatic agents, antioxidants, UV stabilizers, flame retardants or processing aids like defoamers, rheology control agents and so forth. These additives play an important role in regard to surface tension (wettability) and formation of adhesion - especially when they are present on the surface.

In most cases the selection of the polymer type for the part is driven by the required properties like thermal and mechanical properties, resistance against chemicals and last but not least material and processing costs – not by how easily they are to bond. Thermosets often have chemical groups that allow good wetting and reaction with the adhesive. Most of them have a chemical composition similar to or even identical to that of typical reactive adhesives like epoxies or polyurethanes. All of this suggests a better bondability, which is partly the case.

In the group of thermoplastics, both those with and without functional groups are found. Plastics like polypropylene (PP) and polyethylene (PE) are due to the very similar tendency of hydrogen and carbon atoms to attract electrons non-polar (fig. 3) and are characterized by a low surface energy of max. 30 mN/m. This results in a poor wettability and a lack of interaction with corresponding groups of adhesives, which at the bottom of the line makes it difficult to bond to. Replacing one hydrogen atom at every second carbon atom in PE by a chlorine atom leads to polyvinylchloride (PVC). With the chlorine atom having a much higher tendency to attract electrons PVC is somewhat polar (fig. 3)

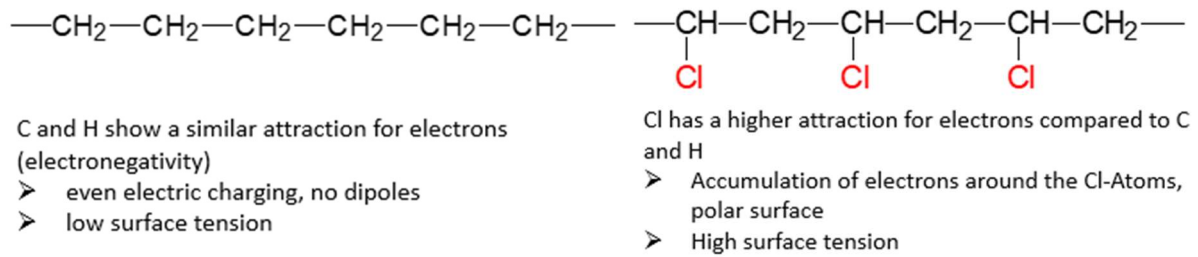


Fig 3: Molecular structure of Polyethylene (PE), left and Polyvinylchloride (PVC), right

and characterized by a significantly higher surface tension of about 40 mN/m which improves wettability. The polar groups are capable to interact with polar groups of the adhesive and allow the formation of adhesion forces between substrate and adhesive as shown in fig. 2.

Surface tension of selected materials			
	[mN/m]		[mN/m]
Steel	(2500*) / 30 - 60	Polycarbonate	34 – 37
Aluminum	(1200*) / 30 – 50	PMMA	33 – 44
Glass	(290*) / 40 – 70	Polyethylene	31
Epoxy	47	Polypropylene	29
Polyamide 6.6	46	Natural Rubber	24
PVC, rigid	40	Silicone	24
ABS	35 – 42	PTFE (Teflon)	18
* Theoretical value, real values are significantly lower			

Fig. 4: Surface tension of common materials.

The world of elastomers is very diverse and ranges from those based on natural rubber to thermoplastic elastomers, silicones and polyurethanes. From a bonding point of view, they are similar to thermoplastics and thermosets. A big difference lies in the high flexibility, which can lead to relative movements in the adhesive joint and thus to a special mechanical load.

Now it is the case that every component surface has a layer-like structure (Fig. 4). Each of these layers have a specific composition with resulting characteristics that one way or the other might have an effect on bondability.

The **base material** determines mainly the thermal and mechanical properties as well as it's resistance against weathering and chemicals. As just mentioned it has also has an influence on bondability.

The deformed **boundary layer** adjoining the base material consists of the same material as the base material. However, the micro structure differs from that due to the molding process. It may show differences in the polymer structure, like orientation of the individual polymer chains and therefore also

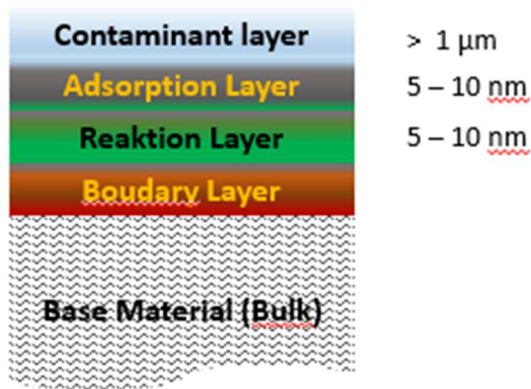


Fig. 4: Schematic structure of a plastic surface

of the functional groups if present, degree of crystallinity, filler and fiber content as well as orientation. Additives contained in the plastic formulation can also accumulate in the boundary during the molding process showing a detrimental effect on bondability.

The **reaction layer** following the boundary layer consists of reaction products of the base material. For metals this are typically oxides but also polymers show such a layer with a compositional difference to the base material.

Indicated already by the name, the **adsorption layer** consists of substances adsorbed from the environment, typically the surrounding air the part has been exposed to. Although the thickness of this layer is usually just a few nanometers the effect on bondability is due to the short range of the adhesion forces quite significant – the adsorption layer prevents the direct interaction between groups of the adhesive and those in the boundary or reactive layer forming adhesion. The adsorbed substances have a much higher binding energy and are therefore harder to remove than the substances in the contaminant layer.

The **contaminant layer** as the outermost layer contains of different substances that have accumulated on the component over time e.g. during storage, transport or up-stream processes. This substances can be dust, but also substances from fumes or aerosols, fingerprints, release agents, etc. For obvious reasons contaminants have to be removed prior to bonding as they are typically not strongly attached to the surface and/or show release properties.

EFFECTS ON ADHESION PROPERTIES CAUSED BY ADDITIVES USED IN PLASTICS, BY AUXILIARY MATERIALS USED DURING MOLDING OR BY INADEQUATE CHANGE OF MOLDING CONDITIONS.

Plasticizer are often used to increase flexibility and durability of plastics. They decrease the attraction between polymer chains to make them more flexible. Typical plasticizer are relatively small molecules and since they are not chemically bound to the polymer matrix they are able to migrate especially when exposed to elevated temperature. In accordance with the principle of nature that differences in concentration should be compensated for as far as possible, the plasticizers show a tendency to migrate into the boundary layer between substrate and adhesive. Thereby, the adhesion-determining interactions are disturbed. Plasticizers can also migrate into the adhesive itself weakening the adhesive's cohesive strength. Both effects result in a reduced load bearing capacity of the bond. Due to the tendency for migration removing plasticizers from the part's surface prior to bonding does not really solve the problem.

A good example is PVC, as explained above PVC has with about 40 mN/m a surface tension which in general indicates good bondability which is true as long as it concerns rigid PVC, not containing plasticizer. If we talk about soft, plasticized PVC usually a good initial strength can be achieved as well but depending on the adhesive used there is a high risk of bond failure after some time due to the described plasticizer migration effect.

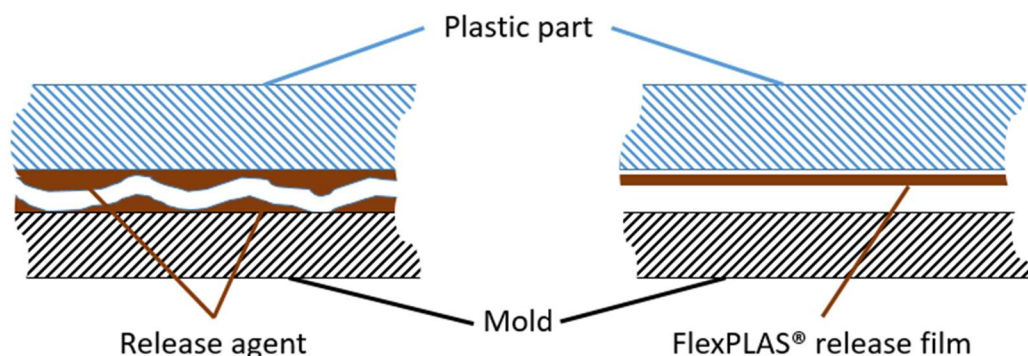
The plasticizer problem can be overcome by plasticizer selection. Polymeric plasticizers show due to the larger size of the molecule a reduced tendency for migration. Selecting an adhesive which tolerates a certain amount of certain plasticizers will further improve bond durability. Best is switching to reactive plasticizer which become part of the polymer network and therefore lose the ability to migrate.

In order to allow for an easy removal of the molded plastic components from the mold, it is usually necessary to use either **internal release agents** or **external release agents**. Release agents are substances used to prevent other materials from bonding to surfaces, e.g. during the molding process of a plastic part. An external release agent is applied to the surface of the mold while an internal release agent is an additive which goes directly into the resin formulation and migrates during the molding process to the interface between part and surface of the mold. In general both types have to be considered as critical for downstream bonding processes as the mold release is not able to differentiate between the surface of a mold and an adhesive. This is expressed in a more or less pronounced adverse effect on the build-up of adhesion between adhesive and part surface.

In recent years, there have been numerous developments to reduce the required proportion of release agent in order to reduce the adverse effect on bonding, and adhesives have been developed that are to some extent able to tolerate certain release agents. In many cases, heat-assisted cure of the adhesive supports the absorption of release agent into the adhesive and thus its removal from the adhesive layer. However, it is difficult to generalize, so that in each individual case it must be checked to what extent under certain process conditions a sufficient adhesion for the respective application is achieved. In many cases release agents must be removed by a pre-treatment prior to bonding the components.

A new most recent development of a separating film that no longer transfer release agents to the component offer new ways for FRP parts manufactured by a prepreg, (vacuum) infusion as well as wet layup processes. The so called FlexPLAS® film [1], is a deep drawable film with differential release properties of its two sides. The film is inserted with the good separating side to the mold surface and therefore allows an easy removal of the part from the mold. The film itself remains on the molded part as a protective film and is only removed immediately before further processing, with no release agent residues remaining on the surface. (fig. 5). An additional positive side effect of such films is the protection of the surfaces of the parts against contamination with anti-adhesive substances. This ensures bonding without any further pre-treatment with high reliability.

A further effect of the use of release agents is on the alignment of polar groups present in the respective polymer. True to the motto "similia similibus solvuntur" (similar substances will dissolve similar substances), the polymer chains align themselves during the molding process in such a way



that

Fig. 5: Release agent distribution during demolding, comparison of the standard process using conventional release agents (left) and the separating film technology (right) /1/

non-polar groups orient themselves towards the also non-polar release agent, i. e. towards the surface. The polar groups, on the other hand, orient themselves away from the surface. As a result, even in the case of plastics containing polar groups, their availability on the part surface is limited. The sum of the resulting adhesion forces is therefore often not sufficient to meet the strength requirements for the bonded part.

Plastics often contain **fillers**, with **fibers** playing a special role. Both short, long and continuous fibers are used. The correspondingly modified plastics are then called fiber-reinforced plastics - FRP for short. Glass, carbon and aramid fibers are mainly used as fibers, but also a wide variety of natural fibers (e. g. flax, jute, cotton) are used. The purpose of the fibers is usually mechanical reinforcement of the plastics, but partly also the adaptation of the coefficient of thermal expansion to other components and adjustment of the direction of dimensional changes by external influences. However, since the fibers improve the mechanical properties of the plastics and the fiber reinforced plastic is often used in high-quality products, this means that the adhesive connections are subject to significantly higher requirements. This concerns both the initial strength, but especially the durability (resistance against alternating mechanical loads, temperature and exposure to moisture and chemicals) as well as the quality and reproducibility of the adhesive bond. As a result, the development and qualifying effort for adhesive joints with FRP substrates is often higher than for "normal" plastic adhesive joints.

Since the fibers primarily reinforce the plastic parallel but not perpendicular to the surface, failure between the fiber layers, i. e. parallel to the surface, can occur already at a relatively low mechanical load. In order to avoid this effect the bond should be located at a sufficient distance from the edge of the part to be joined. It must also be considered that the FRP layer structure may be weakened as penetrated, for example by moisture through unsealed cut edges.

The **parameter of the molding process** has an influence on the surface properties of the part to be produced, in particular on the boundary and the reaction layer, and may influence its bondability. In **injection molding**, for example, the parameters temperature, pressure as well as the number and position of injection points have to be considered. After changing molding conditions, a previously achieved good cohesive bond failure may change to an adhesive failure at a reduced strength level. Particularly in the case of high-strength adhesives it can also lead to a fracture within the boundary or reaction layer of the part, again at a reduced strength level. M. Stege from Volkswagen has reported in a paper about a fender made from PUR-RIM /2/. As shown in fig. 6, the fender consists of a PUR-RIM outer skin and fastening elements, also made of PUR-RIM bonded to the inside of the outer part. The 2-component PUR-RIM raw material contains the reactive resin components, filler and internal release agent as well as various other additives. At a first glance, it was surprising that the good result of lap shear testing (high strength level with a mix of cohesive and substrate failure) could not be achieved consistently in all areas of the part. Good adhesion was achieved in the area close to the A-pillar, but an adhesive failure at a significantly lower strength level was observed in the area of the headlamp. Surface tension measurements in the bonding areas revealed a roughly equal total surface tension, but the polar fraction relevant for the formation of adhesion showed significant differences. In accordance with the different bonding results the polar fraction showed with 39 mN/m significantly higher values in the area of the A-pillar connection than in the area of the headlamp (just 32 mN/m). With just one injection point in the lower area of the A-pillar connection, the

internal release agent migrating to the surface has been pushed by the PUR-RIM material into direction of the headlamp, which results in a significantly thicker layer of release agent and the poor adhesion.

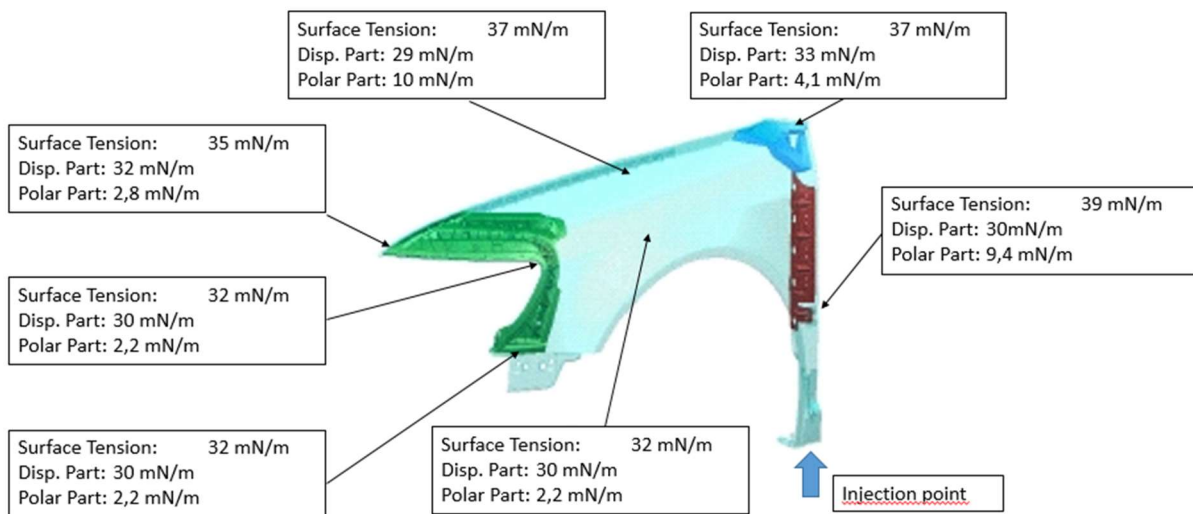


Fig. 6: Results of surface tension measurements on the inner surface of PUR-RIM fender /2/

In the case of **thermoset** materials in which a cross-linking reaction occurs during the molding process, incomplete cross-linking due to a reduction of molding temperature or cycle time may lead to incomplete cross-linking and therefore residual monomers present in the molded part. This can result in a bond failure as shown with fig. 7. In this case bonding of SMC (thermoset glass fiber reinforced polyester) has been done by a 2K-polyurethane adhesive. In order to achieve a short cycle time the cure of the adhesive took place in a heated fixture. The typical failure pattern of the 2K-polyurethane adhesive (green) on SMC after destructive testing is fiber tear. But after a cycle time reduction of the part's molding process the unusual failure pattern at a far reduced strength level was observed. The relatively high amount of residual styrene in the molded part was identified as the cause for the unusual failure pattern. During cure of the adhesive, the parts to be joined are heated from the outside to heat up the adhesive and to accelerate its cure. The heating of the SMC causes the excessive residual styrene to evaporate, which diffuses out of the SMC forming a separating layer between SMC surface and the adhesive and thus prevents the build-up of adhesion forces. In fig. 7, some bubbles can be seen very clearly. In this case, the expanding styrene has actually led to a separation of the adhesive layer from the surface.



Fig. 7: Observed failure pattern on SMC, caused by an incomplete cure and the resulting presence of unreacted styrene which during the heat accelerated cure of the adhesive evaporates, preventing the formation of adhesion and forming the clearly visible bubbles

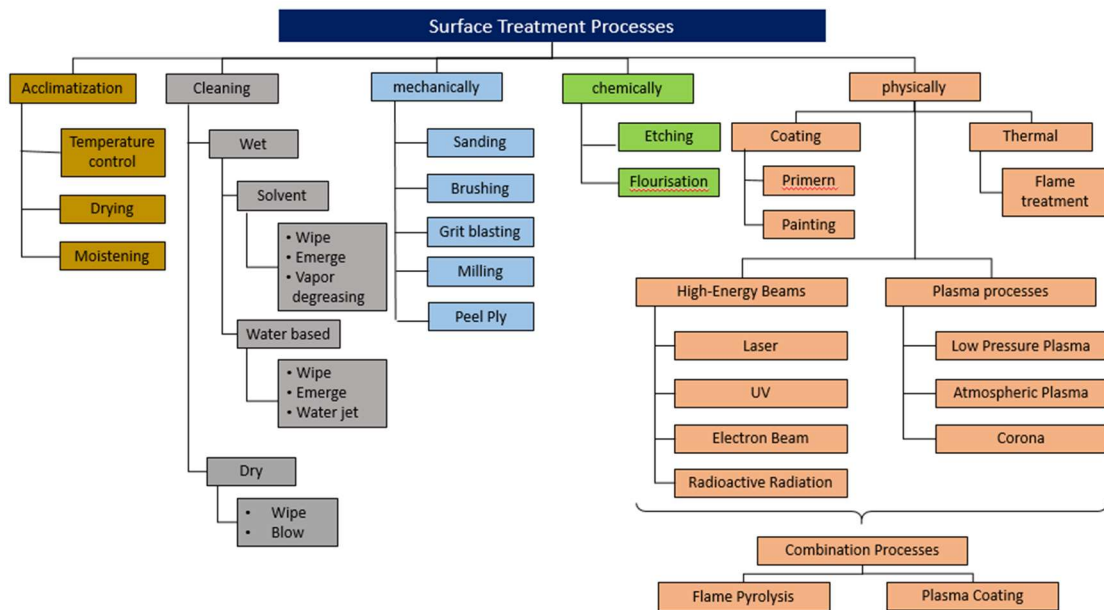


Fig. 8: Surface treatment processes /3/

SURFACE TREATMENT

Most of the technically and economically significant plastics are as a result of the effects described above difficult to bond. But a few thermoplastics mainly PVC, which are soluble or at least swellable in certain solvents can easily be bonded with special solvent-based adhesives. In this process, known as "solvent bonding", the resulting boundary surface layer is the result of a reciprocal diffusion of polymer molecule segments of the surface of the join part and the adhesive molecules to form a substance-locking bond. As this process is limited to a small number of plastics, the bonding of plastics usually requires surface treatment in order to allow adhesive bonding. This ranges from simple cleaning process with the objective to remove surface contamination to chemical or physical processes for activating the surface by incorporating polar groups into the part surface. The common goal is to provide a suitable, defined surface for the subsequent bonding process. Which of the numerous processes (fig. 8) is most suitable must be determined according to the specific application, taking into account the joining component material, the adhesive, the type and quantity of contamination, the requirements placed on the bonded component with regard to its mechanical, thermal and media resistant, the required load-bearing capacity and, last but not least, the costs for each individual case.

A description of the various processes, their fields of application and the details to be observed with regard to the quality of bond would go beyond the scope of this type of article. Regardless of the process the following needs to be observed:

- the effectiveness of the surface treatment needs to be verified. Since the suitability of the respective process depends on the type of contamination, it is recommended to run regular in-process tests, especially if a change in the kind of contamination is possible
- any re-contamination of the surface must be avoided

- it is important to ensure that the plastic is not damaged by the process (e. g. stress corrosion cracking e.g. caused by the solvent used for surface cleaning or as part of a primer),
- any additional contamination caused by the use of unsuitable cleaning materials. Examples are contaminated solvents (denatured ethanol is not suitable, the chemical added for denaturing may not evaporate and, may remain on the part surface causing adhesion problems), used abrasives, chemically treated paper or textile wipes not being lint free, compressed air that is not free of water or oil
- that there is no dragging of contamination – depending on the degree of surface contamination e.g. cleaning wipes or abrasives should be changed sufficiently frequently
- especially in the case of physical processes, an over-treatment with a resulting deterioration of the adhesive properties might happen.

The measurement of surface tension can be used as an in-process test for the effectiveness of surface treatment. This can be done relatively easily using test inks (series of liquids at gradual surface tension values) which are applied as a line on the surface to be tested. If the line of ink stays unchanged for at least 2 seconds without turning into drops, the surface energy of the material is the same or higher than the surface tension of the test ink (fig. 9). Surface tension testing by test inks does have two significant disadvantages. One is that bonding should not take place in the area the test ink has been applied to, it is not a non-destructive test. The second is that it does not differentiate between disperse and polar part of surface tension and may lead to an incorrect interpretation of test result in case there is a different distribution into polar and disperse fractions for the same sum. This differentiation can only be achieved by more complex contact angle measurement. However, this method requires a relatively flat surface, which is not always present on real parts.

A relatively new contactless and non-destructive method for in-line process control of surface quality is the so-called bonNDTinspect® Bondability Test. For testing the surface quality an ultrasonic nozzle wets the surface of the part to be bonded with an extremely fine mist of ultrapure water. In the same process step, a camera takes an image of the droplet pattern before the water evaporates again from the surface to be bonded, quickly and without leaving any residue. The captured droplet pattern is analyzed by an image processing software. The system is using the same physical principle as the contact angle test, but instead of measuring contact angle of a single drop, it analyses the size and distribution of thousands of defined micro-droplets per cm² from the top view, dramatically increasing the statistical significance. It is a reference-based inspection system. This means that the bondability of the parts being tested is determined quickly, reliably and non-destructively by comparing the droplet characteristics with those of a defined good part. /7/

ADHESIVE SELECTION

When selecting an adhesive system suitable for bonding plastics, the parameters already mentioned (fig. 1) and especially the required adhesion and strength level in the relevant temperature range, as well as the ageing behavior and the mechanical properties of the respective parts to be joined must be taken into account. In particular, the strength and deformation of the parts to be joined as well as their dependence on temperature need to be mentioned here. It can be assumed that the adhesive must have an appropriate elasticity in order to compensate for the stress peaks occurring during loading. The modulus of elasticity describing the mechanical properties of the respective plastics is several orders of magnitude lower than the modulus of elasticity of metals. In addition, it continues to decrease as the temperature increases and vice versa, this must be taken into account when selecting the adhesive and in most cases a different adhesive should be used than for bonding a similar metal part.

The number of adhesives offered by the various adhesive manufacturers seems almost unlimited, so it is not easily possible to choose the most suitable adhesive for a particular application. To cover

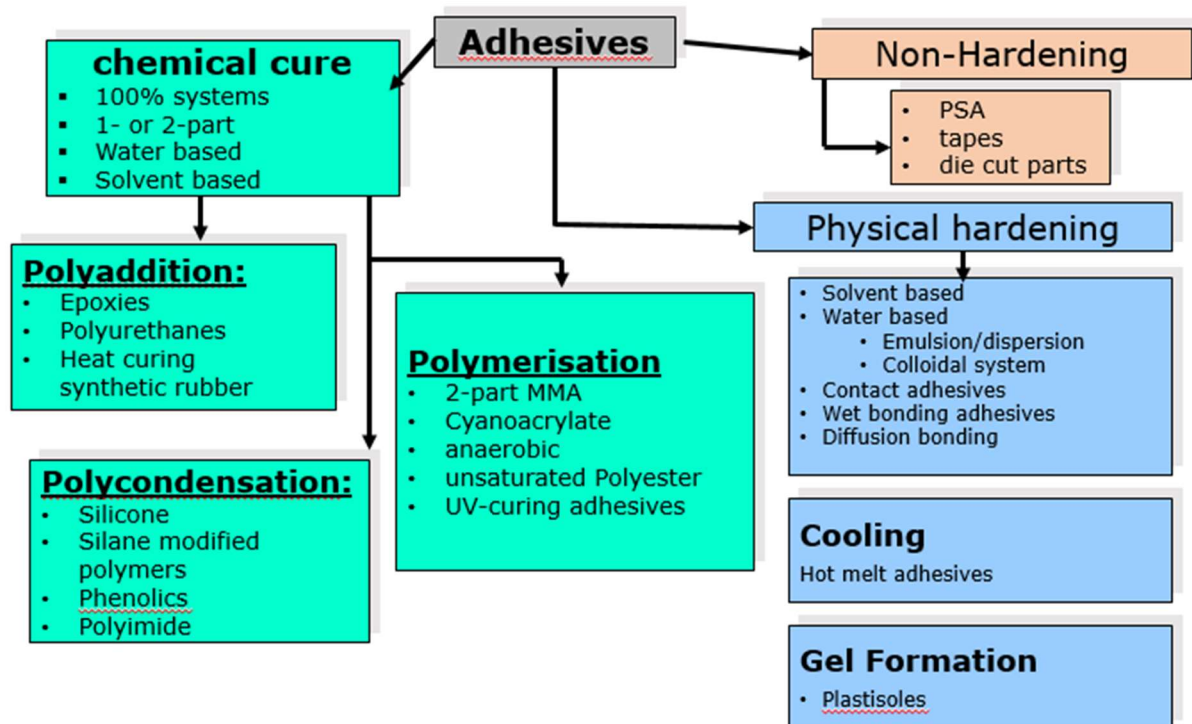


Fig. 9: Classification of adhesives according to hardening mechanisms

individual trading products in this article would only be a snapshot, simply because of new products constantly appearing on the market. Therefore, the different adhesive technologies, characterized by their basic chemistries, are discussed below with regard to bonding of plastics,

Fig. 9 shows a classification of the adhesives according to their hardening mechanisms together with examples of adhesive classes. A basic distinction is made between adhesive technologies which harden by a physical process (e. g. evaporation of a solvent including water, cooling of a melt or gel-ling process) or by a chemical reaction with participation of reactive components contained in the adhesive. In this system, the group of self-adhesive articles (e. g. double-sided adhesive tape) are somewhat special as they offer a fair strength after joining but do not undergo a hardening process.

In case of the physically hardening and non-hardening adhesives, the adhesive polymers are already present in their final size and shape when delivered, whereas in the case of chemically hardening adhesives, these are formed from smaller building blocks in the adhesive joint during the curing reaction. The group of physically hardening adhesives is further differentiated by the respective physical process and the group of chemically curing adhesives by the respective reaction mechanism.

All of the different adhesive technologies do have their pros and cons. Selecting the best is not an easy task and requires not just to consider strength and durability properties but also requirements from the manufacturing process, EH&S properties and last but not least the resulting cost per part, in principle all the individual parameters of fig. 1 have to be taken into account.

TESTING

As required by the relatively new German standard DIN 2304-1: *Adhesives bonding technology – Quality requirements for adhesive bonding processes – Part 1: Adhesive bonding process chain* /4/ published in spring 2016 for all joining technologies and therefore also for adhesive bonding the essential requirement



needs to be met. The verification can be carried out in four ways and needs to be documented:

In /5/ it is explained that the verification of the bonding processes can be carried out in different ways:

- (i.) **Measurements:** Based on the specifications, the load/stress is determined from experiments, calculations, standards, real data or a combination of these. The load/stress limit must be determined by experiment, with accompanying statistics, taking into account the temperature dependence of properties, ageing e.g. due to mechanical stress, exposure to elevated temperature, to light, to chemicals including water and various substances. Combinations of effects need to be considered as well.
- (ii.) **Component testing:** Identifying the load or stress limit can be done by testing a whole system or part of a system under real conditions or under conditions which mimic reality. When testing a part of a system, the mutual interaction between the part-system and whole-system must also be taken into account and it must be verified that this does not falsify the results in an impermissible way. A failure criterion for test evaluation, including an integrated safety factor, must be defined.
- (iii.) **Documented experience** requires that the system has proven itself to be adequate and it shall be demonstrated that the behaviour of the bond under service conditions can be applied to the bond under construction in all relevant terms (e.g. service life, loading conditions, and exposure to ageing effects).
- (iv.) **Combination of 1. - 3.:** The verification by combinations of the aforementioned routes must ensure that all requirements are suitably evaluated and that the individual components are compatible with each other.

Obviously the verification needs to be done stepwise considering all the individual processes:

- part design, especially bond line design according the requirements of adhesive bonding
- material of the parts to be bonded, dimensional tolerances, surface quality and cleanliness as well as surface pre-treatment method if any
- selection of an adhesives considering adhesion properties, mechanical properties, temperature dependency of mechanical properties, durability, dispensing process technology, cure conditions, etc.
- process planning and implementation including preparation of work instructions
- definition of quality assurance measures
- re-work/repair
- ...

For the validation of the entire bonding process in its totality all parameters, including those of up-stream and down-stream processes, including those which are taken place at suppliers or customers have to be considered and defined. It is highly recommended to agree with customers about a list of requirements to be fulfilled by the bonded part and with suppliers on an appropriate specification for all purchased materials and parts taking into account the parameters explained in this article which are or might be of influence to bond quality. Strictly no change should be implemented in production without re-validation assuring that the change does not have any adverse side effect on bond quality.

WORK FORCE QUALIFICATION

A sufficient qualification of the personnel involved in bonding processes is of high relevance, too. Since it is a general requirement for all adhesive bonding processes and would go beyond the scope of this paper, without wishing to diminish its significance, please refer to /4/, /5/ and /6/ for detailed information.

CONCLUSION

When innovative products are launched on the market today, plastics are in many cases involved. They can often only be joined reliably by using adhesive technology - especially when it comes to joining different plastics with each other or plastics with other materials. In order to achieve successful results, the processor should also have a basic knowledge about the plastics to be joined. In order to avoid adverse effects and a potential bond failure knowing how plastics and adhesives influence each other is important, too.

Consideration of the described material properties, both of the plastic and the adhesive, starting with the planning of bond, helps to avoid bonding defects and at the bottom of the line enables those responsible to sleep soundly.

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Dr. Hartwig Lohse offers based on 30 years of experience in the bonding industry independent support on matters relating to adhesive bonding through his consultancy firm KLEBTECHNIK Dr. Hartwig Lohse e.K.

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