

New Proposed Mechanism for the Reaction of Unsaturated Polyester Resins by Alkaline Earth Metal Oxides

by

Kristine E. Eisemon, Plasticolors, Inc.
Jeffrey D. Lewis, Plasticolors, Inc.

Abstract

The thickening of unsaturated thermoset polyester resins by alkaline earth metal oxides in general and magnesium oxide in particular has been the topic of many technical papers over the past thirty five years. The introduction of magnesium oxide into Sheet Molding Compound (SMC) initiated a long and often frustrating investigation of the mechanism of this thickening reaction and the subsequent ability to better control the reaction.

We herein provide a summary historical review of the published theories of the mechanism for the thickening reaction and the pursuit of identifying physical properties which can be correlated to the performance of a thickener. A unique proposal of a mechanism for the thickening reaction is outlined and the evidence to support it is explained. The properties of alkaline earth metal oxides, and specifically magnesium oxide, which are related to thickening performance are also discussed. This discussion is an attempt to understand some of the anomalies that are encountered in everyday formulation and compounding of SMC.

Background

Historically proposed mechanisms, while originating in sound theoretical science, have not been supported by many observations made in the use of magnesium oxide as a thickener for the sheet molding compound (SMC) industry. In addition, efforts to identify a physical or chemical property of the magnesium oxide have been largely unsuccessful in accurately predicting the performance of the raw material. This is not to say that should one be identified, it is the only factor determining performance in an SMC system. There are many other factors that contribute to SMC performance, such as resin selection, moisture content, and additives, which will not be discussed here. The importance of determining

what characteristics magnesium oxide must have in order to thicken SMC is in the possibility to control raw material source. It can also help identify which variables are necessary to control throughout the entire SMC process and formulation. Finally, it may be utilized to identify other raw materials, particularly those that are more robust, that may have the ability to thicken SMC by the same mechanism.

Over the past several years, we have attempted to find evidence to support the accepted mechanism, which involves a simple acid-base reaction, or to elucidate an equally sound mechanism that can account for all of the observations made by the industry with respect to how magnesium oxide thickeners respond to different variables. The most common questions that have been left unanswered by the accepted mechanism are:

- What is the interaction between magnesium oxide and an unsaturated polyester?
- Why do reagent grade and dead burn magnesium oxide fail to thicken SMC?
- If the mechanism involves the dissociation of magnesium oxide, why is magnesium hydroxide less reactive at equimolar ratios of magnesium than magnesium oxide?
- Why does the presence of water accelerate the thickening reaction yet lower the final viscosity?
- Why does the process of spray-drying magnesium oxide yield a less reactive raw material?
- What are the properties of magnesium oxide that determine whether or not it is reactive and can they be measured and controlled to give predictable performance in an SMC formulation?

In the sections that follow, we will briefly review the currently accepted mechanism, propose a new reaction mechanism for magnesium oxide thickening of SMC, discuss supporting evidence, and answer questions where previously proposed mechanisms have failed.

Historically Accepted Mechanism and Physical Properties of Magnesium Oxide

Many have reported either two or four step mechanisms which have been generally accepted by industry, regardless of the anomaly that the mechanism cannot be applied to a broad range of magnesium oxide grades. The two step mechanisms involve the dissolution of magnesium oxide and the formation of either a basic or a neutral salt. The four step mechanism involves dissolution of magnesium oxide and the formation of both basic and neutral salts. In either case, dissolution of magnesium oxide is the first step and the final product is represented in Figure 1¹. We believe the key fault in these theories to be the dissolution step. It is reasonable to assume that if it is the first step, then the choice of magnesium oxide grades should have no effect on the rate of

reactivity. This has not been the case in industrial applications.

Many in the SMC industry have also tried to correlate physical properties of magnesium oxide, or combinations thereof, to the degree of reactivity. Various magnesium oxide samples have been tested for particle size, surface area, loss on ignition, chloride content, moisture content, citric acid reactivity, and many other physical and chemical characteristics. There has been some success in correlating some of those values to specific grades of magnesium oxide for relatively short periods of time. Like the accepted mechanism, these correlations fail when they are applied universally to all grades.

Proposed Mechanism and Supporting Evidence

Our goal was to find a mechanism that could explain the thickening phenomenon in a more universal way. We started by considering the functional groups available in an unsaturated polyester and the types of reactions they can undergo that could possibly lead to an increase in molecular weight. We chose not to consider free radical processes, as they tend to be autocatalytic and lead directly to crosslinking as opposed to simple chain extension. There is sufficient evidence to demonstrate that thickening results in chain extension rather than crosslinking and that associative thickening does not occur. We have completed multiple studies on magnesium oxide thickened polyester by gel permeation chromatography (GPC) and found an increase in molecular weight in the chromatograms. We concluded that the increase was due to chain extension because a crosslinked polymer would be too bulky to come through the filter or would have remained in the GPC column. Only linear or near linear chain extended polymers would have been able to reach the detector. Our results were in agreement with previously published GPC data^{1,2}. Two of the mechanisms we investigated in some depth were the Michael addition reaction and a magnesium ion complex where two terminal acid groups from the polyester acted as bidentate ligands to a four-coordinate magnesium ion, similar to the accepted mechanism product. We were able to find some evidence to support both mechanisms, but the evidence was limited and the mechanisms were as inadequate as previously proposed mechanisms in explaining the behaviors observed in industry.

We completed a thorough literature review of magnesium oxide and its uses in other industries. Many documents referred to magnesium oxide as a selective catalyst for the oxidation of olefins and to a lesser extent paraffins, specifically as a solid surface catalyst or catalyst support^{3,4,5,6}. We took the concept of magnesium oxide as a solid surface catalyst and attempted to work it into a mechanism for SMC thickening and gathered evidence to support or refute the mechanism. A representation of the final form of the mechanism that we propose

for magnesium oxide thickening of SMC is shown in Figures 2 and 3. In Figure 2, the structure is a crystal aggregate of magnesium oxide having a large surface area, and the smaller squares within the unit cells represent lattice defects at the surface. Figure 3 shows a closer view, where the acid group can interact, for example, with the O^{2-} ion vacancies. In this mechanism, the acid group of the unsaturated polyester is chemisorbed to the surface ion vacancies and/or by the strongly basic O^{2-} surface ions on the crystal aggregate. Chain extension is achieved by linking polymer chains with crystal aggregates of magnesium oxide rather than by dissociated magnesium ions as previously accepted. Furthermore, if this mechanism is feasible, then other metal oxides having the same crystal structure, surface chemistry, and types of lattice defects should react with an unsaturated polyester as well.

The first part of our approach was to determine what chemically happens to the polyester when it interacts with magnesium oxide. We began by testing unsaturated polyester with different lots of magnesium oxide known to have small differences in reactivity, reagent grade magnesium oxide, and other metal oxides. The oxides were added to the polyester in equimolar cation ratios and we qualitatively determined whether or not a reaction took place by allowing seventy-two hours for maturation. While the degree of viscosity increase varied, three commercial grades of magnesium oxide, barium oxide, strontium oxide, calcium oxide, and lithium oxide reacted with the polyester. Reagent grade magnesium oxide, beryllium oxide, aluminum oxide, and sodium oxide did not noticeably react. Fourier transform infrared (FT-IR) spectra were collected on unreacted polyester and polyester reacted with magnesium oxide. We observed a broadening and doublet of the carbonyl peak at 1700 cm^{-1} , suggesting some loss of α,β conjugation in the polyester. Additional peaks appeared at 1550 and 1365 cm^{-1} and we observed peak broadening at 1297 cm^{-1} , indicating that the acid group is involved with the thickening reaction.

We encountered some difficulties collecting the ^1H and ^{13}C fourier transform nuclear magnetic resonance (FT-NMR) spectra of the reacted polyester. We were only able to get partial solvation in d_4 -acetone and d_6 -DMSO and the spectra were complicated. We also did not expect to find subtle differences considering that the number of acid groups reacting relative to the total volume of the polymer were very small. In order to study the polymer reaction by FT-NMR properly, we would have had to use solid state FT-NMR, which was not available to us. For these reasons, we chose to continue our evaluation using smaller organic molecules that were functionally similar to an unsaturated polyester resin with the hope of accurately modeling the reaction. We chose a number of carboxylic acid functional compounds and collected FT-IR spectra to determine if they were reactive with magnesium oxide. Those that were reactive were subjected to further testing by ^1H FT-NMR, ^{13}C

FT-NMR, and GPC to confirm an increase in molecular weight. Acrylic acid is most similar to an unsaturated polyester resin in terms of the reactive end group and is a liquid at room temperature, so most of our work was completed using this compound. Of the model compounds that were found to be reactive, the spectroscopic data and GPC data were similar to one another and similar to polyester. In comparing the data from the saturated carboxylic acids with the data from acrylic acid, it was apparent that α,β unsaturation is not a requirement for reactivity. While acrylic acid certainly could not be used to model the kinetics of the reaction, the data showed it to be a reasonable model compound to study the reaction pathway and end product. For these reasons, the remaining discussion of the data will be limited to acrylic acid/metal oxide interactions.

FT-IR spectra were collected using a multi-reflection diamond ATR crystal. The spectrum of acrylic acid as compared to the spectrum of acrylic acid after it has been reacted with magnesium oxide shows a broadening of the carbonyl peak at 1700 cm^{-1} , indicating a slight loss in resonance stabilization. The appearance of peaks at 1550 and 1365 cm^{-1} as well as the broadening of the 1297 cm^{-1} peak are indicative of a vibrational change in the acid group depicted in Figure 4. Acrylic acid was also reacted with the other metal oxides previously tested with polyester and those which were reactive to polyester were also reactive to acrylic acid. On a semi-qualitative level, we measured the exotherm associated with the reaction of each metal oxide and acrylic acid. We found the order of reactivity based on change in enthalpy to be $\text{SrO} > \text{BaO} > \text{Li}_2\text{O} > \text{MgO}$, CaO . The FT-IR spectra showed the same types of changes in the vibrational frequencies of the acid group.

We used a pulsed FT-NMR to collect rotational spectra of acrylic acid in its reacted and unreacted states. Samples were dissolved in d_6 -DMSO and the number of scans ranged from 16 to 64 for ^1H FT-NMR and from 32000 to 64000 for ^{13}C FT-NMR. In both reacted and unreacted species, the $-\text{COOH}$ proton is present. This is significant, as some mechanisms describe deprotonation of this proton as one of the early steps. We have observed that in the case of acrylic acid, as well as other model compounds, that the acid group does not get deprotonated by magnesium oxide, nor by other reactive oxides. The acid proton shifts down field with increasing reactivity of the metal oxide as a result of the proton becoming more shielded. The ^{13}C FT-NMR spectra show that with increasing reactivity, the carboxylic acid carbon shifts up field. Had these chemical shifts been entirely due to hydrogen bonding effects with a polar solvent, they would have been equal among the various oxides. Figure 5 summarizes the chemical shifts as it relates to reactivity.

Once we believed we understood the chemical changes occurring with the polyester and acrylic acid, our focus then turned towards the properties of the oxides themselves. Because magnesium oxide is a group II

alkaline-earth oxide, other alkaline-earth oxides were chosen. Sodium oxide and aluminum oxide were chosen because they represent non-group II oxides, yet are Lewis bases like the group II oxides. Lithium oxide was chosen because it has the closest mass-charge ratio to magnesium oxide. The oxides were qualitatively combined with acrylic acid to determine whether or not a reaction took place. The results and some of the properties of the oxides are listed in Figure 6^{7,8,9}. The metal oxides that are reactive to acrylic acid share the following characteristics; they are group II metals, have a cubic structure, and are strong Lewis bases. One exception is lithium oxide because it is a group I metal. Lithium chemistry is often unique, and we believe that lithium reacts in part because of its antifluorite structure, where the anions are located at the corners of the lattice array. All inorganic crystals have either intrinsic or extrinsic defects or both when formed. Intrinsic defects result from thermodynamics and extrinsic defects result from synthesis and process parameters, which can be controlled to some extent. Cubic structures are prone to as many nine different crystal point defects, and are particularly prone to ion vacancies (Schottky defects), ion displacement into interstitial sites (Frenkel defects), and free electron centers^{7,10,11,12}. The types of point defects and their role in the reactivity of magnesium oxide are discussed in great detail in literature¹². The number and types of point defects is dictated by the temperature at which it is formed and the enthalpic cost specific to the defect type, as described by the law of mass action, which determines the overall disorder of the crystal aggregate¹³. There have been numerous studies regarding the reactive nature of magnesium oxide surfaces^{10-12, 14-18}. Carbon monoxide reductions were carried out over the same set of metal oxides we used for our research and the trends in reactivity were identical¹¹. The authors also noted the structural difference between beryllium oxide and the other group II oxides and reported that its inactivity is likely due to lower basicity and a lack of appropriate reactive lattice defect sites. While each of these references differ in the focus of their study and in the methods used to relate structure to reactivity, all of them credit the lattice defect sites in magnesium oxide as the reactive centers, rather than dissociated magnesium ions. The most common form of direct evidence is electron paramagnetic resonance spectroscopy (EPR) and the most common indirect method is the use of surface probe molecules.

We attempted to utilize surface probe techniques in order to measure the magnesium oxide surface basicity of different grades. We passed t-butyl alcohol vapor through columns, each packed with different grades of magnesium oxide, as is done in inverse gas chromatography (IGC). We then took the resulting magnesium oxide and measured the FT-IR spectrum by using a diffuse reflectance infrared fourier transform spectroscopy (DRIFTS) module. We found a positive correlation between reactivity and the intensity of the sharp peaks at

2980 cm^{-1} and 1220 cm^{-1} , illustrated in Figure 7. Normalization of the absorption to the surface area of the sample being tested yields a more accurate quantitative result. Other surface probe molecules can be used to investigate the chemical nature of the magnesium oxide surface which may provide a better correlation. Direct evidence is desirable, but requires more sophisticated techniques which are unavailable to us. The two most important tools for further research are x-ray photoelectron spectroscopy (XPS) and EPR spectroscopy. XPS is important because it provides a direct surface analysis by detecting the photoelectron energy that is characteristic of the chemical state (oxidation and bonding) of an atom at the surface. EPR can locate atoms in interstitial sites of the magnesium oxide crystal structure by detecting the change in unpaired electrons' spin magnetic moment in differing local magnetic fields within the sample. With these techniques, we may also be able to directly study the magnesium oxide/polymer interaction in real time. Now that instruments are commercially available, IGC may be a viable and more cost effective technique to utilize in testing magnesium oxide prior to introduction in SMC.

Discussion

Utilizing the mechanism we have proposed, we can address the observations made by industry that have traditionally been unexplained by other mechanisms. We can describe the interaction of polyester and magnesium oxide as an acid-base chemisorption of the terminal acid group on the polyester with crystal aggregate lattice defect sites, particularly anion vacancies at corners, steps, and kinks of the crystal surface. The planar surfaces are not expected to react to any significant degree^{3,10}. A three dimensional representation of different faces of a cubic structure is shown in Figure 8. Here, steps and kinks are illustrated for several Miller index crystal faces and are known to be more reactive than terrace surfaces³.

Other mechanisms have relied on the dissociation of magnesium oxide, but if they were correct, reagent grade and dead burn magnesium oxide should react with polyester as well as commercial grades. In addition, magnesium hydroxide at equimolar ratios of magnesium should react to the same degree as magnesium oxide. In terms of the mechanism we have proposed, there is no dependence on dissociation and the reasons for the inactivity of reagent grade and dead burn magnesium oxide become clear. Reagent grade magnesium oxide is commonly made by the smoke method where magnesium metal is reacted with oxygen, resulting in a very pure product with few defects. Our data shows that reagent grade magnesium oxide reacts with acrylic acid and with polyester, but in the case of polyester, it is to a negligible degree with respect to commercial grades. Some lattice defects are available on the reagent grade magnesium oxide and given the number of acids groups per gram of acrylic acid as compared to the number of acids groups

per gram of polyester, this is not a surprising result. It is the relatively few numbers of lattice defect sites that renders this form of magnesium oxide useless for SMC thickening. Dead burn, or sintered magnesium oxide also lacks a considerable number of defects, however for different reasons. Magnesium oxide is thermally activated at temperatures from 400 to 800°C, however the presence of water vapor during this process leads to gross structural changes and sintering, effectively destroying the active defect sites¹¹. We believe that magnesium hydroxide is less reactive because it has a completely different structure than magnesium oxide, although may contain a small number of the lattice defect sites at the surface, depending on how it has been processed. It is equally possible that magnesium hydroxide thickens polyester by a different mechanism.

The role of free water and humidity in the thickening mechanism has been particularly elusive to industry users. Many have reported that the presence of water causes the initial rate of thickening to increase and the final viscosity to be lower than SMC that is kept in a dry environment. The kinetics of the reaction of water with polyester and magnesium oxide are not fully understood and was not the focus of this study. It is well known that when water is added to an SMC system without a thickener present, some level of thickening will take place. We believe that initially, water and magnesium oxide thicken polyester on a competing and possibly additive basis, although the mechanisms are not the same. This can account for the higher initial rate. Water likely increases the molecular weight through the hydrogen bonding of polymer chains. However, water can behave as an acid or as a base. Water as an acid, can occupy the same lattice defect sites on the magnesium oxide as the acid groups on the polyester, leaving fewer sites available for the thickening reaction, and resulting in an observed lower final viscosity. Though these observations can be rationalized by this mechanism, the evidentiary support is not available at this time and the role of water remains a topic for future study.

We were provided with an experimental lot of magnesium oxide that had been spray dried. In testing the reactivity in an SMC system, we found it to be reactive, but to a lesser degree than other commercially available products. This product's reactivity can also be rationalized by the mechanism we have proposed. As mentioned earlier, magnesium oxide can be thermally activated, but it can be mechanically activated as well. Mechanical processing, such as ball milling and vibrational milling, of magnesium oxide has led to an increase in lattice defect sites, specifically centers of oxygen defect electrons as measured by EPR spectroscopy and rates of dissolution in acid¹⁴. It also stands to reason that these lattice defects are not only on the surface of the crystal aggregate, but throughout the aggregate as well. Mechanical activation is accomplished in part by exposing a number of internal lattice defects by increasing the surface area. Spray drying magnesium oxide eliminates

a critical step in producing a highly reactive product, that of mechanical activation.

Conclusions

It is clear that the production of magnesium oxide can be controlled and measured to yield an optimum number of crystal defects. Thermal activation, mechanical activation, water vapor, pressure, and starting materials all play a crucial role in determining the final number and types of defects found. It is equally clear that these variables must be controlled by the suppliers of magnesium oxide. However, the costs of such controls may be prohibitively expensive for it to be practical. We have illustrated a few techniques that show great promise in evaluating and predicting the reactivity of magnesium oxide by indirect measurements. These techniques offer a more cost effective approach to the qualification of magnesium oxide. It is important to remember that the reactivity of magnesium oxide is only one variable related to the performance of SMC thickening. Quantitative measurements of the reactive lattice defect sites give us the upper limit of that particular sample's reactivity. Each component in an SMC formulation that can occupy one of those lattice defect sites ultimately reduces the final degree of conversion to higher molecular weight species, particularly water. The use of these techniques may also provide a means to evaluate other metal oxides or combinations of metal oxides that could prove to be more robust in the SMC process.

Future Work

The specific role of water in the reaction mechanism, including its kinetic effects, requires extensive investigation. It is also unclear whether the polyester remains chemisorbed onto the magnesium oxide crystal lattice or if the lattice defects serve as catalysts for a polymerization reaction. In either case, it is clear that the relative quantity and types of lattice defects are the key to magnesium oxide reactivity. The utilization of this reaction mechanism has led to the development of techniques that are used for the qualification and prediction of thickener performance in SMC systems. A discussion of this development and its practical use in the SMC industry will be presented at a future date.

Figures

Figure-1

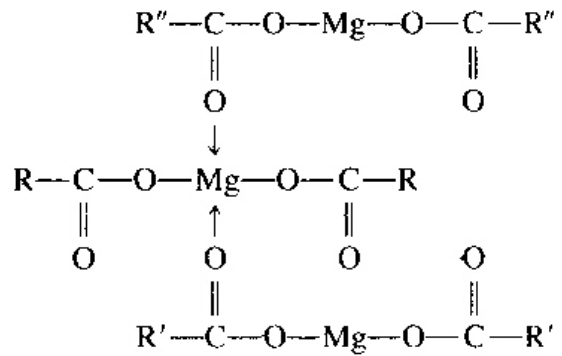


Figure-2

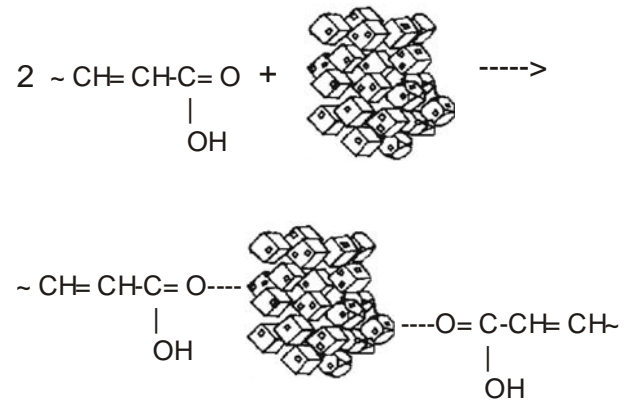


Figure-3

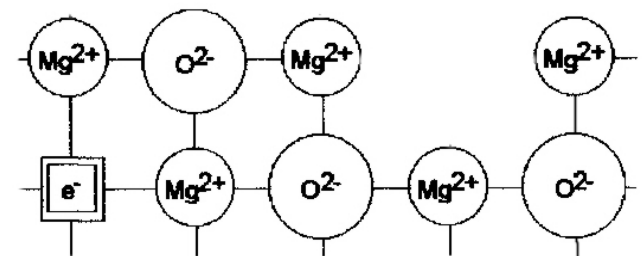


Figure-4

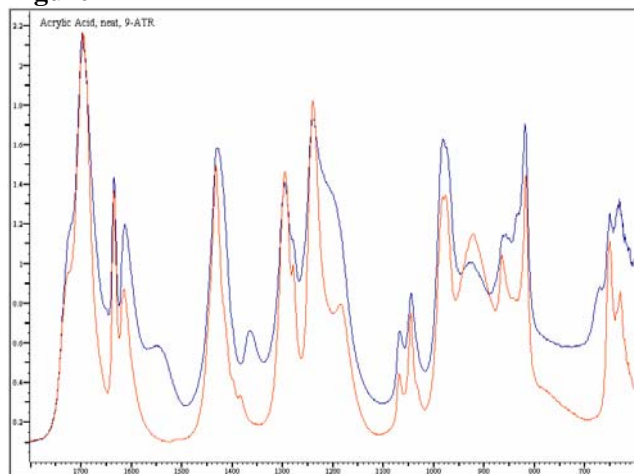


Figure-5

	Relative Reactivity	¹ H NMR Shift	¹³ C NMR Shift
Acrylic Acid		12.38 ppm	167.11 ppm
MgO (reagent)	Low	10.89 ppm	167.77 ppm
MgO (commercial)	Medium	9.31 ppm	168.11 ppm
Li ₂ O	High	7.96 ppm	169.85 ppm

Figure-6

Oxides	Group	Lewis A/B	Crystal Structure	Reactive to Acrylic Acid?
MgO (commercial)	II	Strong Base	Cubic	yes
MgO (reagent)	II	Strong Base	Cubic	yes
BeO	II	Acid & Base	Hexagonal	no
BaO	II	Strong Base	Cubic	yes
SrO	II	Strong Base	Cubic, HCP	yes
Al ₂ O ₃	III	Acid & Base	Cubic	no
Na ₂ O	I	Strong Base	Cubic	no
CaO	II	Strong Base	Cubic, HCP	yes
Li ₂ O	I	Strong Base	Cubic	yes

Figure-7

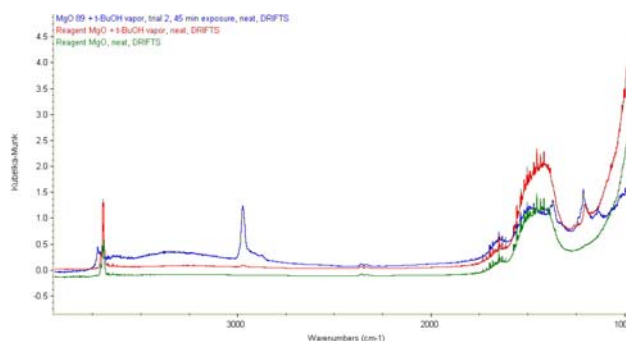
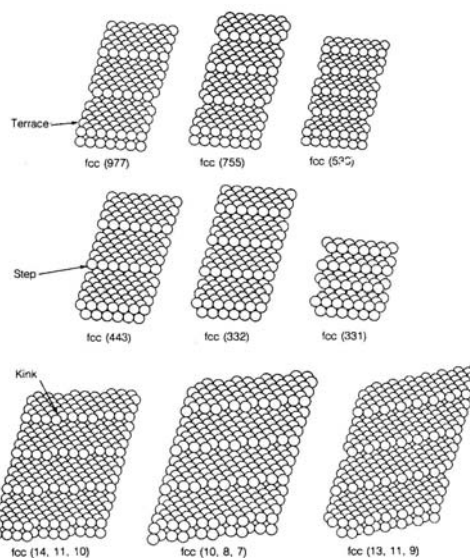


Figure-8



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Citations

1. Kia, H., *Sheeting Molding Compounds*, Hanser/Gardner Publications, Inc., Cincinnati, **1993**.
2. Ludwig, C., Collister, J., *34th Annual Technical Conf., Reinforced Plastics/Composites Institute, SPI*, Session 24-C, **1979**.
3. Gates, B.C., *Catalytic Chemistry*, John Wiley & Sons, New York, **1992**.
4. Choudhary, V.R., Mulla, S.A.R., Uphade, B.S., *J. Chem. Technol., Biotechnol.*, **1998**, 72, 99-104.

- 5 Thoms, H., Epple, M., Reller, A., *Solid State Ionics*, **1997**, 101-103, 79-84.
- 6 Ito, T., Wang, J. X., Lin, C.H., Lunsford, J.H., *J. Am. Chem. Soc.*, **1985**, 107, 5062-5068.
- 7 Cotton, F.A., Wilkinson, G., *Advanced Inorganic Chemistry*, John Wiley & Sons, New York, **1988**.
- 8 Shriver, D.F., *Inorganic Chemistry, Second Edition*, W.H. Freeman, New York, **1994**.
- 9 WebElements™, the periodic table on the WWW, Copyright 1993-2003, Mark Winter, University of Sheffield and WebElements Ltd, UK,
<http://www.webelements.com/webelements/index.html>
- 10 Abbet, S., Riedo, E., Brune, H., Heiz, U., Ferrari, A.M., Giordano, L., Pacchioni, G., *J. Am. Chem. Soc.*, **2001**, 123, 6172-6178.
- 11 Morris, R.M. and Klabunde, K.J. *Inorg. Chem.*, **1983**, 22, 682-687.
- 12 Woodruff, D. P., *The Chemical Physics of Solid Surfaces, Volume 9, Oxide Surfaces*, Elsevier, Amsterdam, **2001**.
- 13 Smyth, D. M., *The Defect Chemistry of Metal Oxides*, Oxford University Press, Oxford, **2000**.
- 14 Jost, H., Braun, M., Carius, Ch. *Solid State Ionics* **1997**, 101-103, 221-228.
- 15 Giamello, E., Murphy, D., Paganini, M.C., *Colloids and Surfaces*, **1996**, 115, 157-170.
- 16 Stefanovich, E.V. and Truong, T.N., *J. Chem. Phys.*, **1995**, 102, 5071-5076.
- 17 Itoh, H., Utamapanya, S., Stark, J.V., Klabunde, K.J., Schlup, J.R. *Chem. Mater.*, **1993**, 5, 71-77.
- 18 Anshits, A.G., Voskresenskaya, E.N., Kondratenko, E.V., Maksimov, N.G., *Catalysis Today*, **1995**, 24, 217-223.

Glossary of Analytical Techniques

GPC: Gel Permeation Chromatography is a special case of liquid chromatography where the separation of molecular weight fragments is achieved by passing a polymer solution through a series of columns containing packing over a gradient of pore sizes. The hydrodynamic volume of the fragment determines whether it will travel through a pore of a specific size or bypass the pore.

FT-IR: Fourier Transform Infrared Spectroscopy is a vibrational spectroscopy technique where the chemical bonds in a molecule absorb infrared light, causing changes in the vibrations of those bonds. Different types of bonds vibrate or stretch at characteristic frequencies, allowing for the identification of the groups present in the compound.

FT-NMR: Fourier Transform Nuclear Magnetic Resonance Spectroscopy is a rotational spectroscopy technique

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where a magnetic field is applied to a molecule, aligning the magnetic moment of the hydrogen protons, in the case of ¹H FT-NMR, or the carbon 13 isotope, in the case of ¹³C FT-NMR, either with or against the applied magnetic field. The amount of energy required to flip a proton or carbon 13 isotope to the less stable state against the applied magnetic field is measured, providing information about the electronic environment surrounding a given proton or carbon 13 isotope. This provides structural information about the molecule.

ATR: Attenuated Total Reflectance is a type of FT-IR where the spectrum is obtained by reflecting the infrared source off the surface of a compound. The use of a multibounce crystal provides constructive interference, yielding greater overall absorption in the spectrum.

EPR: Electron Paramagnetic Resonance Spectroscopy, also known as ESR, Electron Spin Resonance Spectroscopy, measures the absorption of microwave radiation by unpaired electrons, such as those found in free radicals and metal complexes, in an applied magnetic field. It is used for the determination of electronic composition and to study electronic relationships between molecules.

IGC: Inverse Gas Chromatography is a special case of gas chromatography where the sample of interest is packed in the column, rather than passed through a standard column. A reactive probe gas is pulsed through the column and the quantity of the probe gas entering the system and exiting the system is measured. The difference is the measurement of the number of moles of probe gas that have adsorbed to the sample of interest in the column per unit surface area. Careful choice of probe gases can identify specific types of crystal lattice defects.

DRIFTS: Diffuse Reflectance Infrared Fourier Transform Spectroscopy is a type of FT-IR where the spectrum is obtained by reflecting the infrared source off the surface of a rough surface, which results in the diffusion of the light. The DRIFTS module collects the diffused light from all angles and refocuses them to the detector, giving a single additive spectrum.

XPS: X-Ray Photoelectron Spectroscopy, also known as ESCA, Electron Spectroscopy for Chemical Analysis, is a technique where the application of x-rays under ultra high vacuum results in electron emission from surface and near-surface atoms. This provides surface and near-surface compositional details, including an approximation of the oxidation states of those atoms.

Authors:

Kristine E. Eisemon has been the Analytical Chemist at Plasticolors, Inc. for seven years. Her current position includes R&D and supervision of Analytical Services. Ms. Eisemon holds a BA in Chemistry from Hiram College and is nearing completion of an MS in Chemistry from Cleveland State University.

Jeffrey D. Lewis is the Laboratory Manager for Plasticolors, Inc. Mr. Lewis has been with Plasticolors for over 30 years and has performed duties as Technical Service Manager, Color Laboratory Manager, and Quality Control Supervisor. His current position is responsible for research and development, analytical services, and manufacturing scale-up and prototyping. He holds a BS from Kent State University.