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Epoxy Resin Crystallization

Introduction

If you have ever used an epoxy resin, you have likely experienced crystallization. Crystallization shows up as cloudiness, free floating crystals, crystal masses, or as a completely solidified mass. It is the same process that causes honey to crystallize at home.

The crystallization phenomenon is possible in all epoxy resin and hardeners. It is the phase change of a material from a liquid to a solid crystalline state. The main ingredient of most epoxy resins is a solid material at room temperature. Exposure to extreme cold, temperature cycles and other factors may induce crystal growth and revert the materials back to their natural solid state.

Crystallization is difficult to predict or eliminate entirely. It happens without warning and may only affect part of a given lot of material. While it may be only an inconvenience with two-component systems, it can cause major problems with one of the components. Understanding the factors believed to influence crystallization and the methods of dealing with it can turn this problem into merely an annoyance.

Causes

Epoxy crystallization occurs similarly to other crystal growth. High purity, low viscosity, impurities, extreme cold, and temperature cycles all increase the probability of occurrence. Usually, the presence of a “seed” initiates the crystallization process.

Here are some of the factors that contribute to the crystallization process in epoxy resins:

High Purity

A high purity resin has been stripped of all chemical by-products and contaminants and falls within a given range of molecular weights. The presence of by-products and a wide range of molecular weights serve to broaden the temperature transition range between liquid and solid. High purity resins have a narrow temperature range during which they transform from liquid to solid. An analogy is pure water which transforms from liquid to crystalline solid (freezes) at 0ºC. Yet, with the addition of another chemical (ethylene glycol), the water freezes at much lower temperatures. The closer a high purity resin gets to the point where it changes from a liquid to a glassy crystalline solid state, the greater the chance minute crystals will start to form. These crystals act as seeds and, in combination with other factors, can rapidly change the liquid to a solid.

Low Viscosity

Low viscosity resins are very low in molecular weight and short chained. The lower the viscosity, the easier the liquid epoxy can move and orient itself around seed crystals. High molecular weight, high viscosity materials are longer chained and less prone to crystallize. Storing a “seed” free liquid at low (0ºC) temperatures will slow molecular motion and impede crystal formation and growth.

Impurities

Impurities, usually minute particulate matter, can often act as “seeds” in unfilled systems, initiating the formation of resin crystals which then continue to propagate. Fillers rarely initiate crystallization due to their large size and high content. Actually, they often act to inhibit crystallization.

Extreme Cold

While cold does impede growth by slowing movement, extreme cold (-40ºC) accelerates crystal formation once seed crystals have formed and, if low enough, can cause complete crystallization by itself.

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Temperature Cycles

Temperature cycles as little as 20-30ºC can create a vicious circle that is the most common cause of crystallization. Once the material is warmed, molecular motion is enhanced allowing liquid epoxy to orient itself around "seed" crystals. Subsequent exposure of an "oriented" material to cold temperature will then accelerate crystal growth. Once started, the crystallization typically goes to completion resulting in a solid mass. The temperature fluctuations that occur between daytime and nightime temperatures can initiate and/or accelerate the crystal growth process. These can occur during transit, while sitting on a loading dock or production floor.

Solutions

Crystallization in base resins and two component formulations is a major inconvenience but not an imsurmountable problem. Heating these materials overnight at 65ºC easily reverses the phenomenon. Like honey, all the crystals must be completely melted as any microscopic unmelted crystals will act as "seeds" and cause the crystallization to return in days. Along with the gentle heating, the material should also be stirred and the container sides and bottom scrapped to assure all crystals have been melted and the heat is being evenly distributed. If crystals reappear, merely apply heat and remelt.

One component systems should not be heated as product damage or curing may occur. One component systems which have crystallized within the stated shelf life and the recommended storage conditions, may have to be returned for replacement.

Controlling and monitoring shipping and storage conditions is critical for minimizing temperature fluctuations. Good housekeeping is another factor as container spouts, spigots and closures must be kept free of resin build-up to prevent crystal formation here as well.

While epoxy compounders and end users have heightened their awareness to crystallization, it still remains very difficult to accurately predict or eliminate. If crystallization remains a recurrent problem, products less prone to crystallize may have to be evaluated as alternatives.

Conclusion

There are not hard and fast rules of crystallization. However, refrigeration limiting temperature fluctutations and simple warming to melt the crystals represent easy ways of dealing with this common phenomenon.

Note

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