

Look into a tunnel freezer and see technology that can be used for cryogenic liberation of composite materials.

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**COOLING**

# RECYCLING COMPOSITE MATERIALS USING Liquid Nitrogen

**Composite parts such as wires, circuit boards or plated pieces present unique challenges to recyclers due to the mix of materials. Cryogenic processing using liquid nitrogen can help recyclers recover more materials for reprocessing.**

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Today, everything from household products to complex industrial parts is recycled, and the use of recycled materials in the manufacturing sector continues to increase due to environmental and economic benefits. Worldwide, the recycling industry generates about \$200 billion in revenue. Ten percent of this revenue is spent on research and development for new technologies that enable the recovery and reuse of materials.

One of the recycling industry's challenges is recovering composite parts — products that are made up of different materials bonded together. Parts made from a polymer and a metal such as electrical cables, vibration isolators, oil-well seals and metal tracks with rubber pads contain materials that can be valuable if they are recovered and recycled (figure 1). For effective recycling, however, composite parts first must be separated into their individual components using

cost-effective processes that will not deteriorate the value of the components to be recycled.

Current technologies such as incineration, pyrolysis and chemical separation can be expensive and unfriendly to the environment. Additionally, these techniques may not be able to recover the highest-value material from the part. Cryogenic liberation using liquid nitrogen (LIN) is one such process that can recover high value materials from multi-component parts.

## Principles of Cryogenic Liberation Using Liquid Nitrogen

The cryogenic liberation process uses liquid nitrogen to cool composite parts to cryogenic temperatures (down to -320°F [-196°C] if necessary) to facilitate the separation of individual components. Because each component will have a different coefficient of thermal expansion, each component

will contract at a different rate as it is being cooled. Additionally, elastomeric components will embrittle as they are cooled down past their glass-transition temperatures, making them more susceptible to fracturing. In many cases, the differential contraction — as a result of different coefficients of thermal expansion in combination with embrittlement — creates enough stress to break the physical bonds between the different components, leading to their separation. (Coefficients of linear thermal expansion for some common materials are shown in table 1.)

In general, polymer materials have much higher coefficients of thermal expansion than metals. This suggests that almost all composite materials made up of metal bonded to rubber or plastic can be successfully separated using cryogenic liberation. However, in practice, some combinations of polymers and metals have proven to be difficult to separate even at liquid nitrogen temperatures.

# CRYOGENIC SYSTEMS

While physical properties allow for some predictability of separation efficiency through cryogenic cooling, experimental testing of composite materials often is still required to determine if the cryogenic liberation process would be a viable solution versus alternative recycling techniques.

## Experimental Testing

The evaluation of cryogenic liberation for a composite material begins with a laboratory-scale test where part samples are immersed in liquid nitrogen to determine the effectiveness of separation. Samples are cooled to various cryogenic temperatures to identify the optimal operating temperature for the process. The effect of cooling rate on the separation efficiency also is analyzed to determine the optimal cooling method to be used during pilot testing.



**FIGURE 1. Many types of insulated wires can be treated with cryogenic liberation using liquid nitrogen. Other materials that can be cryogenically treated include circuit boards, metal/rubber weather stripping, plated plastics and composite parts.**

Calorimetry then is performed on the samples to determine specific heat capacity, which is subsequently used to obtain an initial estimate of liquid nitrogen consumption for the process.

The next step to determine the feasibility of the cryogenic liberation process typically involves using

a commercially available piece of cryogenic equipment to conduct pilot testing. During pilot testing, special attention is given to confirm the separation efficiency at a large scale as well as to optimize the overall liquid nitrogen consumption. (This includes houseline and equipment cool down, liquid nitrogen consumption during production mode and liquid nitrogen consumption during idling.) Depending upon desired production rate, degree of separation and operating temperature, several cryogenic cooling systems are available on the market. The most commonly used systems are liquid nitrogen immersion freezers and liquid nitrogen tunnel freezers.

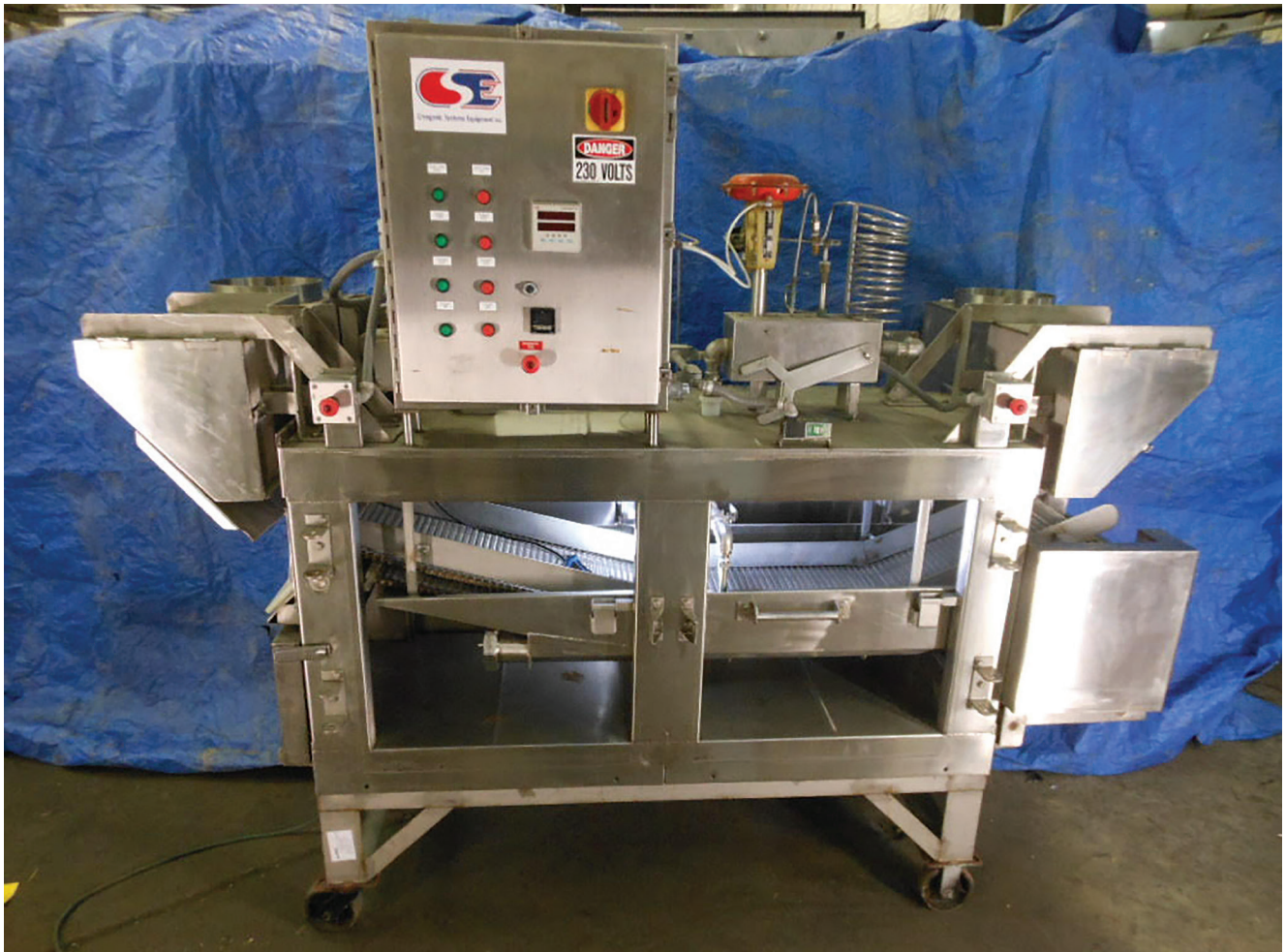
**Immersion Freezers.** Liquid nitrogen immersion freezers provide refrigeration to parts by conveying them through a bath of liquid nitrogen. Many types of immersion freezers are available on the market and a typical system is shown in figure 2. In immersion freezers, the longer the part is held in contact with the liquid nitrogen bath, the colder the part will be when it exits the freezer. Hence, residence time is an important parameter that can be regulated through conveyor belt speed.

Consider, for example, a composite part that requires cool down to an average temperature of -100°F (-73°C) to be separated into its individual components. Based on calorimetry

**TABLE 1. Linear Temperature Expansion Coefficients of Common Materials**

Product	Linear Temperature Expansion Coefficient ( $\alpha$ )	
	( $10^{-6}$ m/(m K))*	( $10^{-6}$ in/(in R))*
ABS (Acrylonitrile Butadiene Styrene) Thermoplastic	73.8	41
Aluminum	22.2	12.3
Copper	16.6	9.3
Ethyl Vinyl Acetate (EVA)	180	100
Hastelloy C	11.3	6.3
Iron, Pure	12	6.7
Nylon, General Purpose	72	40
Nylon, Type 6, Cast	85	47.2

\*) m/m = meter per meter or in/in = inches per inches



**FIGURE 2. Immersion freezers provide refrigeration to parts by conveying them through a bath of liquid nitrogen. In immersion freezers, the longer the part is held in contact with the liquid nitrogen bath, the colder the part will be when it exits the freezer. Residence time is an important parameter that can be regulated through conveyor belt speed.**

measurements, the calculated residence time in liquid nitrogen is 15 sec. Assuming the length of the liquid nitrogen bath is 5 ft, then the conveyor belt needs to operate at a speed of 5/15, or 0.3 ft/sec (20 ft/min) to allow all the parts to reach the required exit temperature.

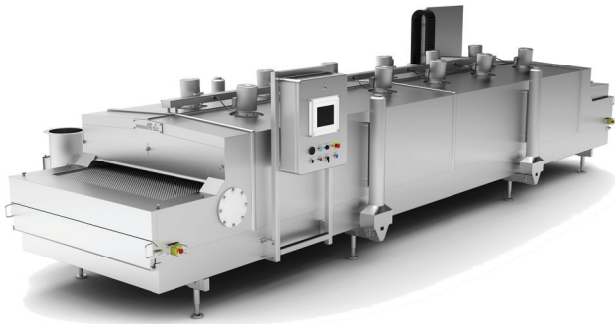
Conveyor belt loading, liquid nitrogen bath length and conveying speed are three independent variables that need to be balanced to achieve the desired production capacity with the required residence time. It is important to highlight that the liquid nitrogen bath needs to be sufficiently deep for the part to be completely immersed within the bath. Full immersion results in more uniform heat transfer throughout the part, which provides more efficient separation.

Depending upon the specific heat capacity of composite parts, typical operating liquid nitrogen consumption ratios for an immersion freezer ranges from 1:1 to 3:1 (weight of liquid nitrogen consumed per weight of parts processed).

**Tunnel Freezers.** Liquid nitrogen tunnel freezers

are another technique that can be used for cryogenic liberation of composite materials. In this method, instead of immersing parts in liquid nitrogen, the material is sprayed directly onto the parts as they move along the length of the tunnel freezer (figure 3).

One of the main advantages of tunnel freezers over immersion freezers is the potential to significantly reduce overall liquid nitrogen consumption. During immersion freezing, only the latent heat of vaporization of liquid nitrogen – the amount of energy absorbed by the liquid nitrogen to evaporate to nitrogen gas – is used, which is equivalent to 86 BTU/lb (199 kJ/kg) of refrigeration. By contrast, tunnel freezers are designed to extract more of the cooling capacity of liquid nitrogen by taking advantage of both the latent heat of vaporization of liquid nitrogen and the specific heat of cold nitrogen gas – the amount of energy absorbed by the cold nitrogen gas to warm up close to room temperature. By utilizing both of these cooling



**FIGURE 3. Liquid nitrogen tunnel freezers are another technique that can be used for cryogenic liberation of composite materials. In this method, instead of immersing parts in liquid nitrogen, the material is sprayed directly onto the parts as they move along the length of the tunnel freezer.**

regimes, a typical tunnel freezer is able to provide about 130 BTU/lb (302 kJ/kg) of refrigeration.

To better illustrate this example, imagine you have determined that you need to remove precisely 30 BTU (31.7 kJ) from a single composite part to achieve adequate separation. Using an immersion freezer, 1 lb of liquid nitrogen will allow you to process 86/30, or approximately 2.9 parts. Alternatively, using a tunnel freezer, 1 lb of liquid nitrogen will allow you to process 130/30, or approximately 4.3 parts. This benefit becomes significantly more apparent at larger scales.

The tradeoff for increased liquid nitrogen consumption efficiency is a larger footprint and increased complexity to optimize the process. The footprint is typically bigger because the average rate of heat transfer along the overall length of a tunnel freezer is slower than in an immersion freezer. Therefore, longer residence times are required, which means that the length of the tunnel has to increase or the belt speed has to decrease to achieve the same degree of cooling as in an immersion freezer.

In addition to the three main variables described above for immersion freezers, tunnel freezing also has to take into account the use of fans to maintain a uniform temperature along the length of the tunnel and to optimize the heat transfer rate between the parts and the cold nitrogen gas. All of these variables need to be balanced to achieve the desired production rate with the required cooling capacity.

In our experience, cryogenic liberation processes are first optimized using immersion freezers and later upgraded to tunnel freezers to take advantage of the increased liquid nitrogen efficiency.

Many current technologies for recycling composite parts can be expensive to use, unfriendly to the environment and may not be successful in recovering the highest-value materials.

Cryogenic liberation is one cost-effective process that

uses liquid nitrogen to cool parts to cryogenic temperatures to break the physical bonds between individual components. Simple laboratory testing can be performed to determine the feasibility of cryogenic separation of materials. Recyclers should consult a company with knowledge and experience in the area of cryogenic technology to help identify the optimal equipment to be used. An experienced manufacturer also can help define the necessary operating parameters for recovering high value materials from multi-component parts. **PC**

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