

# Considerations about Filling Materials Influence upon Deflection of Epoxy Resin under Cryogenic Conditions

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*The paper presents the methodology of determining the plasticity of some composites specimen through bending test. The equipment is presented both for room temperature testing and under cryogenic conditions. Particularities of the measurements under cryogenic conditions are specified. The influence of talc concentration in the composite material is presented.*

*Key words: composite materials, deflection, cryogenics.*

Composite materials are a class of engineering materials with special technical and scientific features. They are widely applied, due to their properties and qualities, in fields such as: civil and industrial buildings, electronics, road railway and naval transportation, aviation and aerospace industry etc. Development of the manufacturing parts from plastic and composite materials represents an alternative for the industry which is in a full process of restructuring.

Composite materials structure can be easily modelled so that the products can satisfy any requests. The proper selection of the nature, size, geometry and proportions of the reinforcing materials, as well as the nature of auxiliary materials and the fabrication technology, enables the production of a wide and detailed range of new composite materials.

Frequently, these materials are used at low and very low (cryogenic) temperatures, due to their purpose. The manner in which they perform at these temperatures is influenced by the fabrication formula that was used. The slightest modification in the formula is responsible for obtaining different values for mechanical characteristics. For optimisation the design of the composite material it is necessary to determine the mechanical characteristics both at ambient and cryogenic temperatures.

The cryogenic engineering, as a branch of non-conventional technologies, examines the development and use the techniques and technologies of producing and application of low and very low temperatures. Regardless of any subsequent application, the first step to be made is the study of the properties of the respective materials in cryogenic conditions comparatively with those at ambient temperature.

The development of cryogenic science in the last years was possible due to the great number of industrial applications. Understanding how cryogenic temperatures influence the physical properties as well as mechanical and technological characteristics for materials, give the possibility of using them in devices that work under 120K. The temperatures below these values are called cryogenic temperatures. There are a lot of possibilities to obtain such temperatures by using one of cryogenic fluids presented in table 1 [1].

**Table 1**  
CRYOGENIC FLUIDS PROPERTIES

Lichidul criogenic	T <sub>v</sub> [K]	ρ <sub>l</sub> [ $\frac{g}{dm^3}$ ]	ρ <sub>g</sub> [ $\frac{g}{dm^3}$ ]
<sup>3</sup> He	3,19	58	25,6
<sup>4</sup> He	4,215	125	16,9
H <sub>2</sub> (p)	20,27	71	1,338
D <sub>2</sub> (p)	23,63	161	2,30
T <sub>2</sub> (p)	25,04	257	3,14
Ne	27,09	1236	9,58
N <sub>2</sub>	77,3	809	4,614
CO	81,6	790	4,5
F <sub>2</sub>	85,0	1502	5,63
Ar	87,3	1394	5,8
O <sub>2</sub>	90,2	1140	4,47
CH <sub>4</sub>	111,6	423	1,82
Kr	119,7	2200	8,33

T<sub>v</sub> -vaporisation (or liquefying ) temperature at 1 atmosphere;

ρ<sub>l</sub> -liquid density at T<sub>v</sub>;

ρ<sub>g</sub> -gas density at T<sub>v</sub>.

Generally, all plastic materials increase their strength and decrease their plasticity in cryogenic conditions. For composite materials things are not always the same because of the filling materials used in combination [2]. There is little information about how different filling

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materials influence mechanical characteristics of the composites. In order to use a certain combination for parts used at low temperatures, it is necessary to determine some of the mechanical characteristics of the composite, both at ambient temperature and under cryogenic conditions.

In this paper the authors present experimental researches concerning how the quantity of the filling ingredient, namely talc, influences the mechanical characteristics of a thermosetting material, namely epoxy resin. The equipment that was used for tests is presented and the particularities are discussed. Deflection test was performed at room temperature and under cryogenic conditions, and bending resistance and deflection were determined.

### Testing methodology and equipment

Deflection test at room temperature was performed following the instructions from SR ISO 178: 1998. The equipment used for the similar test under cryogenic conditions has two main parts: the testing machine itself and the cryostat. The machine is a testing universal machine INSTRON 1196 that is equipped with an automatic system for recording the load variation diagram, as an electrical sensing device measures the load. The speed used for bending is 1mm/min and it was kept at the same value during testing. The class of precision of 0.5 ensures an error of exactness of  $\pm 0.5\%$  from the applied load; the relative error of coming back to zero is 0.25% from the maximum limit of measuring, while the threshold of sensitiveness is of 0.05% from the maximum limit of measuring.

The specimen is placed onto the rolls of the bending device shown in figure 1. The punch is fixed in the superior rod tie (fig. 2) of the testing machine. The device is placed inside the cryostat onto the inferior rod tie.

Cryogenic liquid used is nitrogen with the liquefying temperature of 77K. It is poured into the isolated vessel

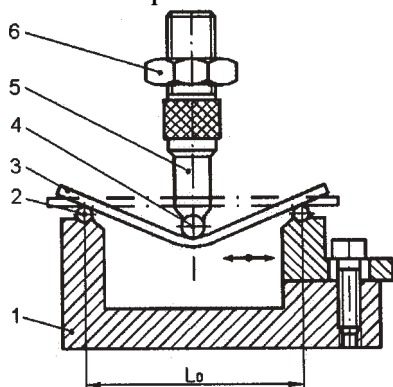


Fig. 1. Bending device; 1 -bending plate, 2 -rolls, 3 -specimen, 4 -punch, 5 - support, 6 -fixing device

from a Dewar container, through the vent pipe and cool nitrogen vapours are obtaining inside. The temperature is measured and at 120K the bending test is performed.

The cryostat is presented in figure 2. It is the modulus type and can be used for traction, compression and deflection measurements [4].

If there is a need of a higher cryostat, it is possible to mount between inferior and superior parts a middle modulus designed and built due to necessities.

### Measurements and results

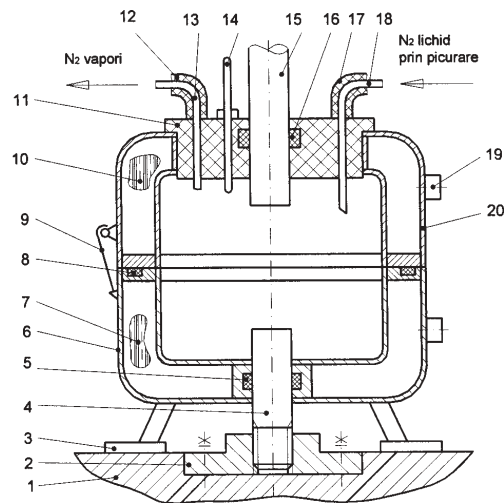


Fig. 2. Modulus cryostat; 1-pressing table; 2-plate; 3-support; 4-inferior tie rod; 5-seal ring; 6-exterior cover; 7 and 10-super isolation; 8-seal ring; 9-butterfly gate; 11-cover; 12 and 17-pipe isolation; 13-vent pipe; 14-thermometer; 15-superior tie rod; 16-seal ring; 18 - supply pipe

The samples were realised from epoxy resin, Dinox type, with talc in different ratio as a filling ingredient, through resin transfer procedure. Polymerisation was performed in oven at 60°C for two hours. In this way 14 samples were realised for each filling ingredient ratio. Samples were realised as required in SR ISO 178: 1998. They have: 80 mm lengths (minim); 10 ± 0.5 mm width; 4 ± 0.2 mm thickness.

Width (b) and the thickness (h) are measured with an accuracy of 0.1 mm respectively 0,002 mm, in 3 points from the calibrated part of the specimen.

Load is very slowly performed at the middle of the specimen and no chocks are allowed. INSTRON machine registers automatically the load variation diagram.

With the measured values (width and thickness) and the obtained ones from registered diagram, bending breaking stress is calculated.

$$R_i = \frac{3 \cdot F \cdot L}{2 \cdot b \cdot h^2} \text{ [MPa]} \quad (1)$$

where:

F - bending load,

L<sub>0</sub> - distance between bearings, [mm];

b -specimen width, [mm];

h - specimen thickness, [mm].

The value of the deflection is determined by using the linear part of load-deflection diagram registered by the machine.

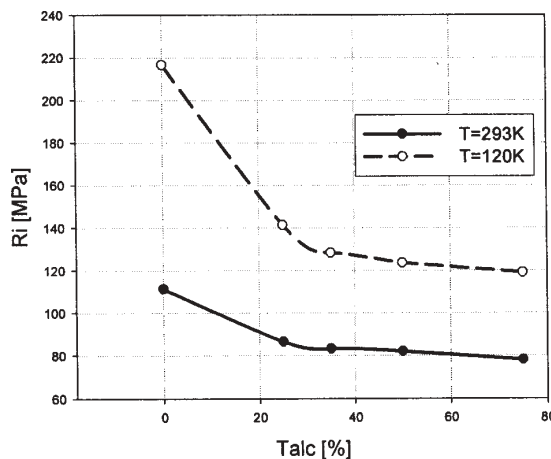


Fig. 3. Talc influence upon bending stress

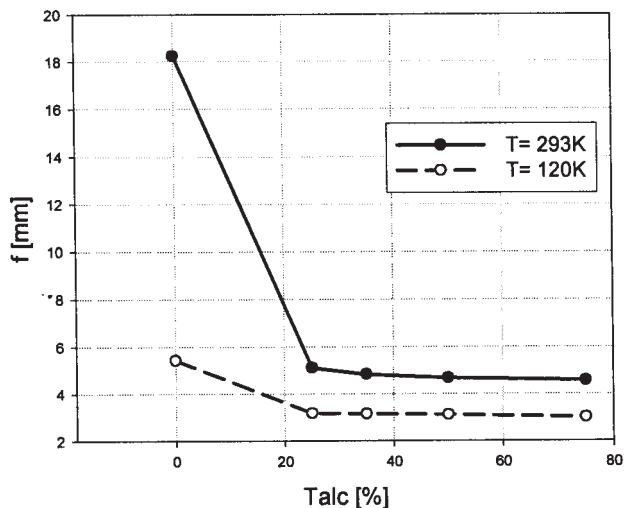


Fig.4. Talc influence upon deflection

First it can be observed that at cryogenic temperature stress resistance is higher than at the room temperature (fig. 3).

It also appears from the diagram that bending stress decreases if an auxiliary material such as talc is incorporated. The slope of the curve is higher in cryogenic conditions so the talc decreasing influence is bigger. Bending stress values at cryogenic temperatures, for specimen with talc in percents higher than 20%, are only a little bit bigger than those obtained for plane resin at room temperature.

The values obtained for greater talc ratio do not vary proportionally with the quantity of the auxiliary material.

The values for deflection are lower under cryogenic conditions as shown in figure 4. That means that the material is more fragile if it is cooled. Only a small amount of talc increases considerably bending material resistance and diminishing its plasticity.

At room temperature the slope of the curve has greater angle than at 120K. The influence of talc is greater at room temperature than under cryogenic conditions.

## Conclusion

Under cryogenic conditions bending resistance of the resin is higher than at the room temperature while plasticity is smaller. It is obviously that a composite material based on epoxy resin filled with talc does not improve bending stress values, no matter which are the temperatures used for tests. Adding an auxiliary material, namely talc, both values vary but with different values for different temperatures. It appears from the diagrams that once the auxiliary material is incorporated, the influence upon properties does not vary proportionally with the quantity. Only a small amount of the ingredient is enough to diminish significantly the measured values. It can be recommended a usage of a recipe with values lower than 20% talc for equipments that work in cryogenic conditions, but only if there is a small bending deflection on it.

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