

# Lithium fluoride material properties as applied on the NIRCam instrument

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## ABSTRACT

Single crystal Lithium Fluoride (LiF) has been base-lined as one of the optical materials for the Near Infra-Red Camera (NIRCam) on the James Webb Space Telescope (JWST). Optically, this material is outstanding for use in the near IR. Unfortunately, it has poor mechanical properties that make it very difficult for use in any appreciable size on cryogenic space based instruments. In addition to a dL/L from 300K to 30K of  $\sim 0.48\%$  and room temperature CTE of  $\sim 37\text{ppm/K}$ , LiF deforms plastically under relatively small stresses. This paper will discuss the heritage of LiF in space-based systems and summarize the mechanical and thermal material data for LiF that is available in the literature. New data will be presented relative to a design limit load for the material so that designers can use this material for space flight applications. Additional new data relative to the cryogenic index of refraction of the material over the near infrared is also provided.

**Keywords:** NIRCam, James Webb Space Telescope, Lithium Fluoride

## 1. INTRODUCTION

The Near Infrared Camera (NIRCam) instrument for NASA's James Webb Space Telescope (JWST) is one of the four science instruments installed into the Integrated Science Instrument Module (ISIM) on JWST intended to conduct scientific observations over a five-year mission lifetime. NIRCam's requirements include operation at 37 Kelvin to produce high-resolution images in two wave bands encompassing the range from 0.6 to 5  $\mu\text{m}$ . In addition NIRCam is used as a metrology instrument during the JWST observatory commissioning on orbit, during the initial and subsequent precision alignments of the observatory's multiple-segment 6.3 m diameter primary mirror. JWST is scheduled for launch and deployment in 2012.

Three optical materials were chosen by the NIRCam optics team to meet the optical performance criteria for the NIRCam instrument. They are Lithium Fluoride (LiF), Barium Fluoride ( $\text{BaF}_2$ ), and Zinc Selenide ( $\text{ZnSe}$ ). This paper gives a brief survey of the material properties of LiF. A significant amount of work has been done over the last 50-60 years to fully understand the mechanical nature of LiF material. The present research has considered papers that discuss almost every aspect of LiF behavior in a myriad of environments. It is not the intention here to present a complete survey of the material, but to offer a survey of those properties that are relevant to the application; a high performance, space-based optical instrument operating at 35K.

Because of the nature of the NIRCam instrument, a complete understanding of the mechanical and thermal properties for LiF is crucial. The authors are unaware of any other space mission that has flown LiF optics of the size and quality that will be present on NIRCam.

## 2. MECHANICAL CHARACTERISTICS

LiF has several characteristics that present challenges for application in space. It has the lowest mechanical strength of all the materials being used for optics on NIRCcam. It has a room temperature coefficient of thermal expansion (CTE) of  $\sim 37$  ppm/K with a  $dL/L$  from 35K to 300K of  $\sim 0.49\%$ . It has a slight propensity to absorb water (0.27g/100g water at 20°C), and a very low apparent elastic limit in its commercially pure single crystal form (11MPa). It also has a tendency toward plastic behavior at stresses well below its published apparent elastic limit.

### Crystal Structure and Growth

Lithium Fluoride has a cubic NaCl, rock-salt structure. For high quality optics, it is grown as a single crystal in a vacuum via the Bridgman-Stockbarger or equivalent method. It is also grown in air via the Kyropoulos method, but this is less common and does not produce as high an optical quality. Both of these crystal growth methods are discussed at length in the literature [1]. The materials most favorable slip plane is the  $\{110\}$  family of planes with a slip direction of  $\langle 110 \rangle$ . There are therefore, six independent slip systems for LiF as there are six  $\{110\}$  planes in a unit cell and one slip direction in each plane.

LiF has a cubic structure and so its elastic behavior is completely described by three compliance constants ( $s_{11}, s_{12}, s_{44}$ ) or stiffness constants ( $c_{11}, c_{12}, c_{44}$ ) where the fourth-order stiffness tensor,  $C$ , is the reciprocal of the compliance tensor,  $S$ . An excellent resource for values of these constants at 4K and higher for many different materials has been compiled in reference [2].

### Anisotropy

As with many single crystal materials, single crystal LiF is not isotropic when loaded mechanically. Elastic modulus and poisson ratio both vary depending on how the crystal is being loaded. Elastic modulus will vary from 85 GPa in the 100 direction to 124 GPa in the 110 direction (See Figure 1).

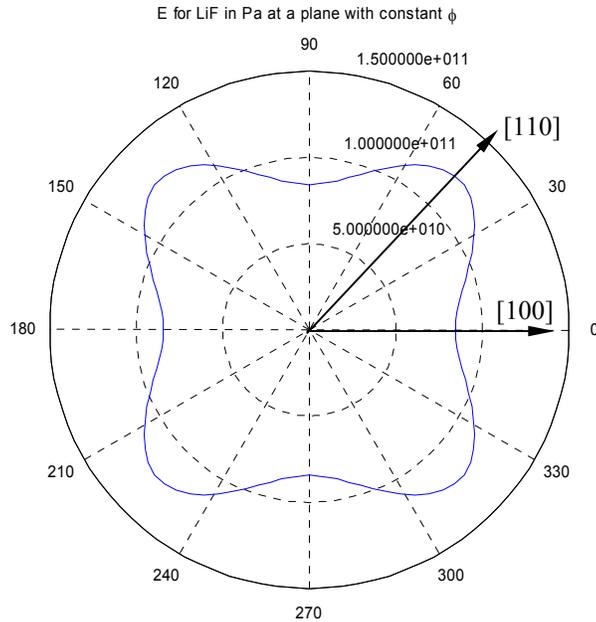


Figure 1: Variation of elastic modulus in Pa with crystallographic loading direction for LiF.

A rough assessment of the anisotropy of a material can be obtained by computing the materials anisotropy factor,  $A$ . This factor is a function of the compliance constants  $c_{11}$ ,  $c_{12}$ , and  $c_{44}$ :

$$A = 2c_{44}/c_{11}-c_{12} \tag{1}$$

An anisotropy factor of unity indicates that the material is isotropic with respect to elastic modulus. A plot of anisotropy factor as a function of temperature for LiF is shown in Figure 2.

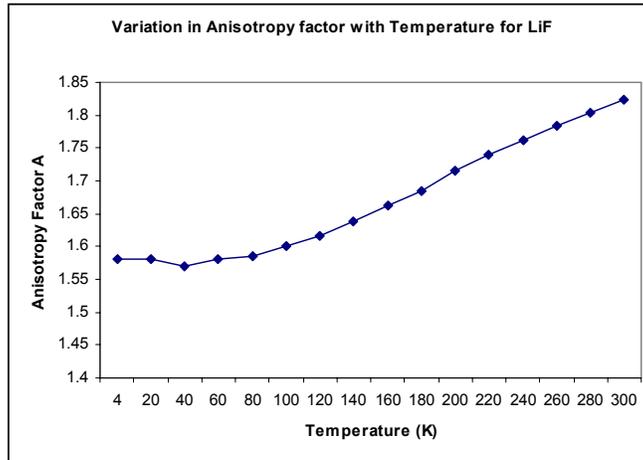


Figure 2: Anisotropy factor as a function of temperature for LiF. Values for stiffness constants taken from [2].

As seen in this figure, elastic modulus anisotropy of LiF increases significantly from cryogenic to ambient temperatures. Other properties can also be anisotropic for some single crystal materials, particularly those that are not cubic, including such mechanically important values as the coefficient of thermal expansion (CTE). This additional anisotropy generally results in further complications in the design of components containing these crystals. Fortunately, LiF does not exhibit properties other than elastic modulus and Poisson ratio that are anisotropic.

### Dislocations and Plasticity

The previous research that has been conducted on dislocations in LiF as they relate to plastic behavior is extensive. Many papers over the last 50 years, including some excellent work by Johnston and Gilman in the late 1950's and early 1960's, have been presented on this topic. For the purposes of NIRCcam, it is important to determine the maximum load that LiF can be subjected to during launch and cool down and still perform adequately on orbit with little or no permanent distortion. Accordingly, a brief review of plastic deformation in LiF is given in the following.

Similarly to glass, single crystal optical materials can also have very high strength and can be brittle. Silicon, for example, has a yield strength of 120 MPa and low fracture toughness of only 0.9 MPa m<sup>1/2</sup> [4]. One of the other materials on NIRCcam, BaF<sub>2</sub>, while not as strong as silicon, also behaves in a brittle fashion when loaded to failure. Contrary to amorphous glass, a single crystal is an ordered group of atoms that form a lattice. If atomic interaction within the lattice is strong, then it is unlikely that slip by dislocation motion will occur very easily. Consequently, the crystal behaves in a brittle fashion. If, on the other hand, the atomic interactions in the lattice are relatively weak for a given family of crystallographic planes, then the crystal may have a propensity to slip on those planes.

The area of research on dislocation movement in single crystal solids is large, and a complete discussion is beyond the scope of this paper.\* For the purposes of this discussion, it is important to note that the density of dislocations in a material, as well as their mobility will determine how the material will deform under load. Purity of the material and fabrication methods play a role as well, but primarily in that they affect dislocation mobility. Since dislocation sources

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\* For good discussions on dislocation theory, see references [5] and [6].

on a surface are a direct result of the way an optic is polished (smoother surfaces generally have fewer dislocation sources) it is important to carefully designate the surface finish and polish on a LiF optic.

The data shown in Figure 3 compare dislocation velocity measured by Johnston and Gilman [3] in polished LiF single crystals with applied shear stress. It can be seen in this figure that below 0.5 kgf/mm<sup>2</sup> (4.9 MPa) dislocation motion is practically non-existent.

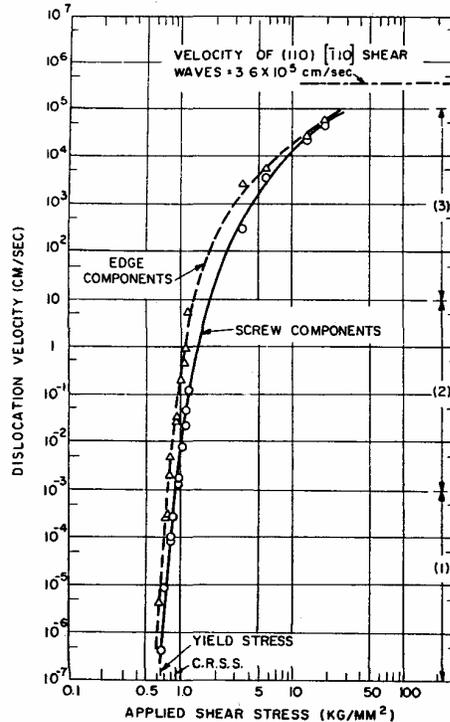


Figure 3: Dislocation velocity as a function of applied shear stress in LiF single crystal (from [3]).

## Creep

Another large area of research related to LiF behavior is in the subject of its creep. A study was undertaken at the University of California at Irvine to determine the ambient temperature creep behavior of LiF single crystals oriented in (100), (110) and (111) directions. The crystals were loaded in an MTS servo-hydraulic test system under compression. Both steady state creep and cyclic creep have been measured, but we will only present steady state data here.

The specimens for this test were cut from a vacuum grown single crystal boule. The ends were polished, but the cylindrical surfaces were ground only. The specimens were 10mm in diameter by 20mm long. The different oriented specimens were put under constant load at 2, 4, 6, 8 and 10 MPa. Creep rates were measured up to durations of six hours as a conservative upper limit for secondary creep rate. These rates are plotted as a function of applied stress on a double logarithmic scale in Figure 4. These data indicate that the creep rates for the C2 <100> orientation are over an order of magnitude greater than those for the other two orientations for the same applied stress. The results of a linear regression for each set of data (C1, C2, and C3) are also shown in Figure 4. Stress exponents were determined from the slopes of the regression lines and are listed in the equations in Figure 4. The estimated stress exponents are all similar and consistent with the value of  $n = 3$  determined by Biberger and Blum [7] for LiF crystals tested at higher temperatures but under similarly low stresses. This value of stress exponent indicates that dislocation climb by the diffusion of vacancies has prominent a role in the creep deformation of LiF for the present conditions. Frost and Ashby [8] have pointed out that impurity effects can alter vacancy concentrations and thus diffusion coefficients substantially. It is also apparent that the coarse ground surface treatment on the sides of the present samples may have resulted in substantially larger vacancy and dislocation densities compared to those for fully polished specimens.

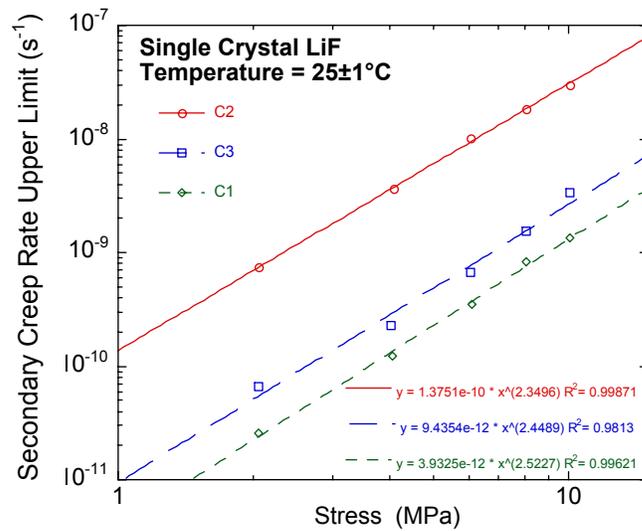


Figure 4: Creep rate after six hours as a function of constant load in LiF oriented specimens. This rate was considered a conservative upper limit for the secondary creep rate.

How these data reconcile with data from Johnston and Gilman is yet to be determined. It appears that the coarse ground cylindrical surfaces provided significant dislocation initiation and resulted in greater creep strains down to the very low applied stress of 2MPa. Further work needs to be conducted with fully polished specimens to fully analyze the role of surface treatment on this behavior.

### Resolved Shear Stress

Slip occurs on the  $\{110\}$  family of planes and along the  $\langle 110 \rangle$  direction in LiF. When a stress is applied to a single crystal, it is important to know how that stress is resolved onto the slip planes and in the slip directions. The *critical* resolved shear stress corresponds to the stress level at which the material will start to slip. In an optical mount, the obvious goal is to keep the resolved shear stress below this value. We can calculate the resolved shear stress in a crystal by considering the direction in which the crystal is loaded with respect to its crystallographic orientation. Figure 5 (from [6]) shows how a tensile load can be resolved onto a shear plane in a material. If  $\phi$  is the angle between the slip plane normal and the tensile axis and  $\lambda$  is the angle between the slip direction and the tensile axis, the component of force  $F$  acting in the slip direction is given by  $F \cos \lambda$ , and the area of the slip plane is given as  $A / \cos \phi$ . Therefore, the resolved shear stress is given by

$$\tau = (F \cos \lambda) / (A / \cos \phi) \quad (2)$$

The value of the critical resolved shear stress  $\tau_c$  is the value at which dislocation motion is initiated in the specimen and the lattice starts to slip.

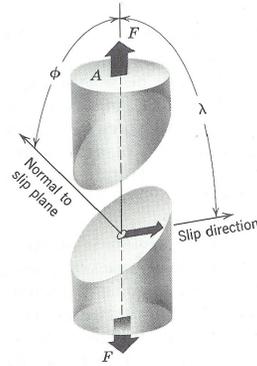


Figure 5: Schematic of the method for calculating resolved shear stress (from [6])

Clearly, this is a value that should be avoided with margin for a space borne optical system. A typical range of values for  $\tau_c$  for various material types over temperature is shown in Figure 6 (from [6]). LiF can be classified as an ionic solid with the NaCl structure, and therefore falls into the wide band of values in the middle of the graph in Figure 4. Johnston and Gilman suggested that an appropriate value for  $\tau_c$  for LiF is  $\sim 5\text{MPa}$ . For NIRCcam, a value (which includes safety factor and margin) of 2 MPa (290psi) is being used.

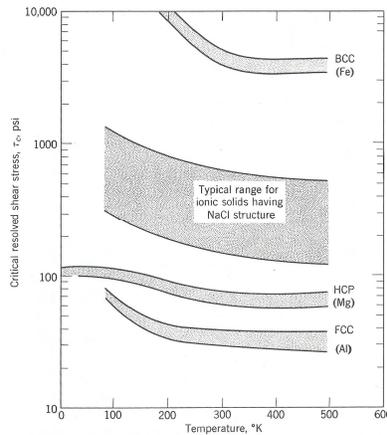


Figure 6: Temperature variation of critical resolved shear stress for several classes of crystalline materials (from [6]).

### 3. OPTICAL CHARACTERISTICS

#### Cubic Crystals and Refraction

Refraction in single crystal LiF is isometric due to the symmetry and electronic structure of the lattice. That is, no double refraction occurs as in crystals such as sapphire.

#### Index of Refraction over Temperature and Wavelength

As mentioned in the introduction, the waveband of interest for NIRCcam is 0.6 to  $5\mu\text{m}$ . While room temperature index data for these wavelengths can be found in the literature, cryogenic index of refraction data has until recently been sparse. Users of IR materials at cryogenic temperatures needed to use Tropp or Sellmeier models to predict index at a given temperature and wavelength. In the case of NIRCcam, it was felt that errors in these model predictions would be too large to accommodate the overall wave front error budget for the instrument.

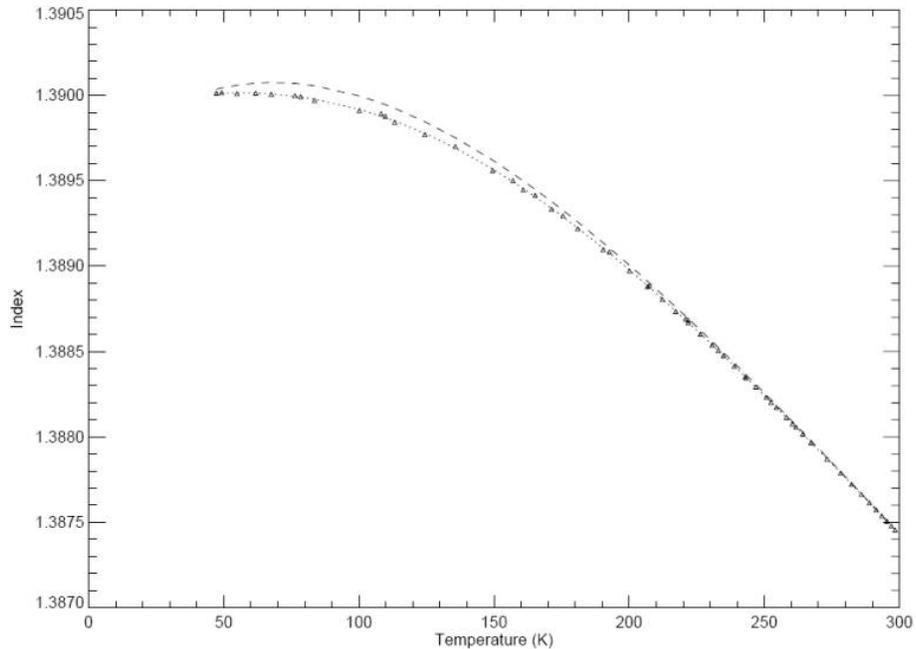


Figure 7: Variation of refraction index over temperature for LiF at a wavelength of  $1\ \mu\text{m}$  (the dotted line represent the Tropf model, the solid line is measured data)

This gap in reported data is rapidly being filled, however, by work that has been performed on the Cryogenic High Accuracy Refraction Measuring System (CHARMS) at the NASA Goddard Space Flight Center by Leviton and Frey. Detailed information on this testing has been provided in [9] and [10]. A representative plot of index vs. temperature is shown in Figure 7. Actual data is accurate to within 3 parts in  $10^{-5}$ .

### Color Correction

Lithium Fluoride is, in almost every respect, a weak structural material. So why not use another material with better mechanical properties that transmits over the same waveband? A detailed answer to this question is out of the scope of this paper, but this has been addressed in detail by Jamieson in [11]. In summary, LiF in conjunction with  $\text{BaF}_2$  and ZnSe provides good focus control over a wide waveband (apochromatic correction). Certainly, there are other combinations of lenses that accomplish a similar result over various wavebands, but these three materials working together were found to be far superior in the NIRCcam range of operation ( $0.6\ \mu\text{m}$  to  $5\ \mu\text{m}$ ).

## 4. THERMAL CHARACTERISTICS

### Heat Treatment

Johnston and Gilman [3] state that dislocation motion was significantly affected by the manner in which the crystal boule was cooled in the manufacturing process. They saw reductions in hardness of the material as high as 80%. Today, almost all single crystal material that is of optical grade is grown via vacuum Bridgman-Stockbarger method. This method cools the boule very slowly as internal stresses are a concern. It is important to be aware however, that heat treatment of the boule can cause significant changes in properties. See Figure 8.

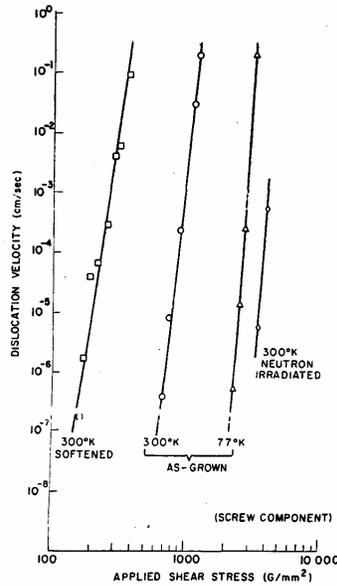


Figure 8: Variation in shear stress for equivalent dislocation velocity in hard and soft LiF crystals (from [3])

### Thermal Shock

In his paper on thermal shock [12], D.P.H. Hasselman offered a method to determine how capable a material will be to resist thermal shock without fracture. Physical properties of the material are taken into account, and depending on the rate of cooling, a parameter  $R$  is defined. There are several different variations of  $R$  depending on cooling rate, and for NIRCam the applicable value is  $R'$  which is the figure of merit for mild heating.  $R'$  can be obtained using

$$R' = \frac{S(1-\nu)k}{\alpha E} \quad (3)$$

where  $S$  is mean strength,  $\nu$  is Poisson's ratio,  $k$  is the thermal conductivity,  $\alpha$  is the CTE, and  $E$  is the Young's modulus. Calculated values of this figure of merit are given in Table 1.

	Expansion Coefficient $\alpha$ E-6/K	Young's Modulus E GPa	Thermal Conductivity k W/mK	Poisson's Ratio $\nu$	Mean Strength S Mpa	$R'$ W/m
LiF	37.0	85	14.2	0.27	11	37
BaF2	17.0	65	7.1	0.31	27	120
CaF2 (Hot Forged)	18.9	76	10	0.28	55	276
Ge	6.1	103	59	0.28	90	6085
Sapphire	5.3	344	36	0.27	300	4324
ZnSe	7.6	70	16	0.28	50	1083
ZnS (standard)	7.0	74	19	0.29	100	2604

Table 1: Hasselman figures of merit for thermal shock. E for LiF is in the 100 direction.

A low value indicates a poor ability to resist shock. LiF is relatively weak in this regard as shown in Table 1. Consequently, significant temperature gradients in a LiF lens for a space borne optic must be avoided. Further work needs to be performed to know exactly what an acceptable value for maximum temperature gradient should be for a

polished LiF optic. For NIRCcam, a value of  $\sim 7\text{K}/100\text{mm}$  maximum gradient is the design criterion which has been used. This gradient value is well above that predicted for the most conservative cool down profiles on NIRCcam [13].

## 5. MOUNT FOR NIRCAM

The NIRCcam instrument is launched warm ( $\sim 300\text{K}$ ) and operates after a passive cool down on orbit at  $35\text{K}$ . The mount for the LiF optic is designed to limit the stress induced on the optic during launch and keep the optic centered during cool-down and operation. In order to accommodate the NIRCcam system requirements, a 12 pad circumferential flexure design was utilized (Figure 9).

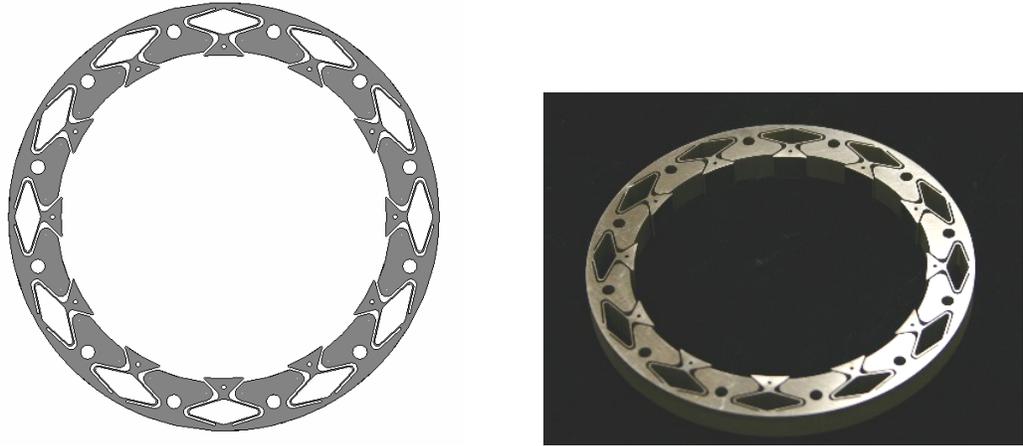


Figure 9: The diamond flexure lens cell design and manufactured titanium lens cell hub

Each of the twelve flexures are shaped in the form of a diamond. The diamond flexures include pads that interface the optic and are contoured to match the radius of that optic. In order to spread out the load at each pad, a compliant, low modulus material is placed in between the metal flexure pads and the lens. This prevents any metal to crystal contact, which could result in local deformation of the crystal. Each of the 12 radial pads is preloaded to allow appropriate limited motion during launch. The pre-load must however be low enough to prevent the material from yielding during launch and cool-down, when mechanical and thermal stress will be at there highest.

In the axial direction, the optic is loaded into the base of the lens cell via 12 flexures around the circumference of the lens. Each flexure has the same compliant, low modulus material below it that was used for the radial pads. These pads also act to spread out the pre-load and prevent metal to crystal contact (see Figure 10).

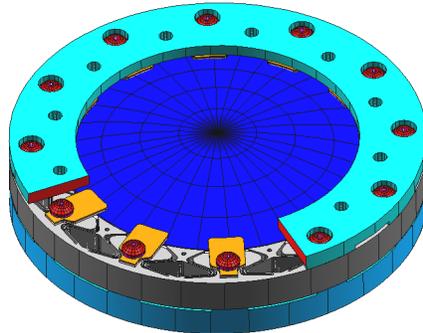


Figure 10: A cutaway view of the NIRCcam lens mount showing the axial pads

A detailed discussion of this mount, including analysis and performance testing has been presented in [13].

## 6. CONCLUSION

A summary of the properties of LiF relevant to the NIRCam instrument has been presented. Mechanical, thermal and optical properties have been reviewed, and the mount used for the NIRCam optics briefly discussed. The plastic behavior of this material under load presents the mechanical team on NIRCam with a number of challenges. These challenges have been addressed through a great deal of effort by the team to produce a mount that will accommodate a LiF lens and perform on orbit. To the knowledge of the authors, no other space-based system has utilized LiF lenses of this size before.

## 7. ACKNOWLEDGMENTS

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