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HIGH PURITY SILICA REFLECTIVE HEAT SHIELD DEVELOPMENT

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MR. BLOME: I would like to very briefly describe to you the development program that we have with NASA Ames on the high purity reflective heat shield material.

As summarized on Figure 6-43, we selected the SiO_2 material primarily because it is very highly reflective in the wavelength band of interest. Also, it is shock resistant, has good ablation characteristics, and we feel that the cost would be competitive with other materials.

The major factor, as I discussed, is the fact that it is highly reflective in the correct wavelength band. The factors that influence the reflectance, we feel, are purity and morphology. By morphology, we mean the internal nature of the particles, the shape, size, and void size.

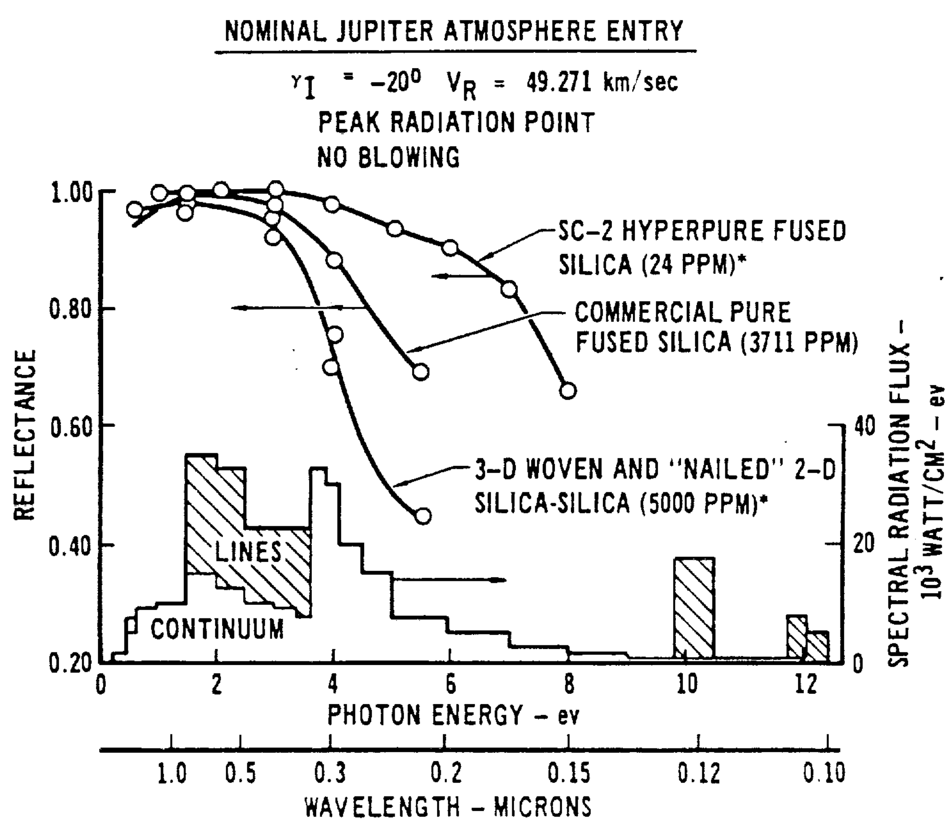
I would like to thank Aerotherm for the use of their spectral flux data which I have plotted on Figure 6-44 for a twenty-degree entry into the Jupiter atmosphere. I said that purity is very important, and this slide primarily addresses the purity effect. We have determined reflectance for three different purity levels of material. The five thousand ppm material, which we feel is quite impure has an SiO_2 binder which contains most of the impurities. Commercially pure, slip cast material, which was Glasrock, has about a 3,700 ppm. These are the total metallic ion concentrations.

This top curve on the figure is for a slip cast part, similar to the one I passed around. In the fabricated state, it has approximately twenty-four ppm. We start with a material that has about 1 ppm total metal impurity ions.

What we did next is to take this spectral flux and integrate it with the three reflectances for these three different purity levels. This is shown on Figure 6-45, which shows how much energy

- REASONS FOR SELECTION
  - HIGHLY REFLECTIVE IN THE CORRECT WAVELENGTH BAND
  - GOOD ABLATION CHARACTERISTICS
  - EXCELLENT THERMAL SHOCK RESISTANCE
  - READILY FABRICATED TO FULL SIZE AT REASONABLE COST
- FACTORS THAT INFLUENCE REFLECTANCE
  - PURITY
  - MORPHOLOGY

Figure 6-43. Silica Selected as the Reflective Heat Shield Material



*TOTAL METALLIC ION CONTENT ESTIMATED FROM REFLECTANCE DATA

Figure 6-44. Purity Affects Reflectance

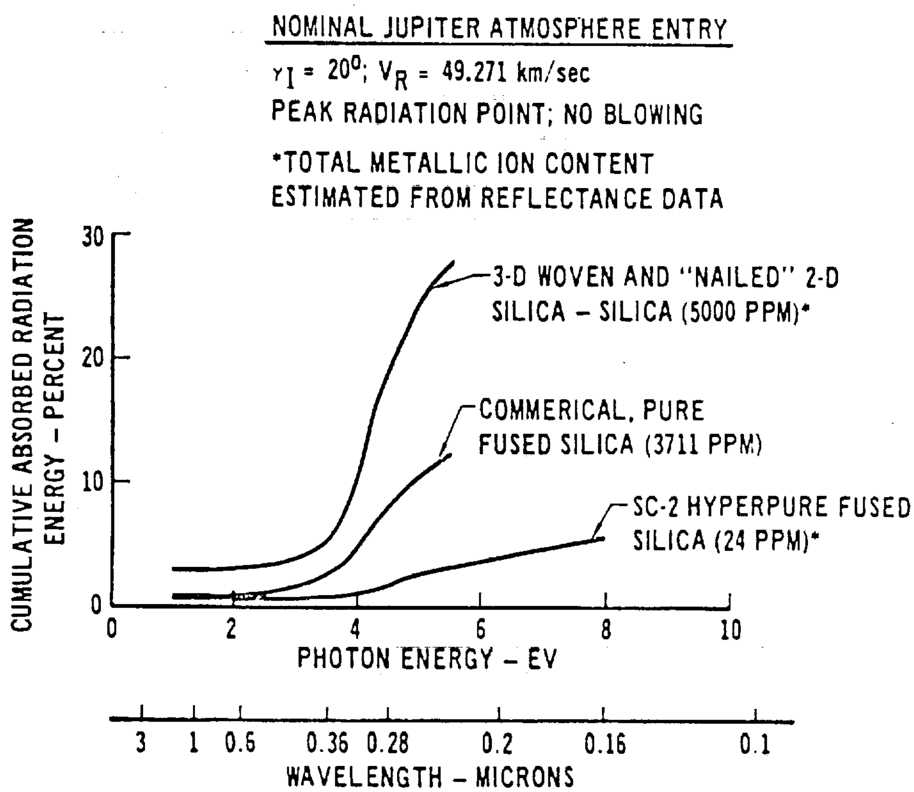
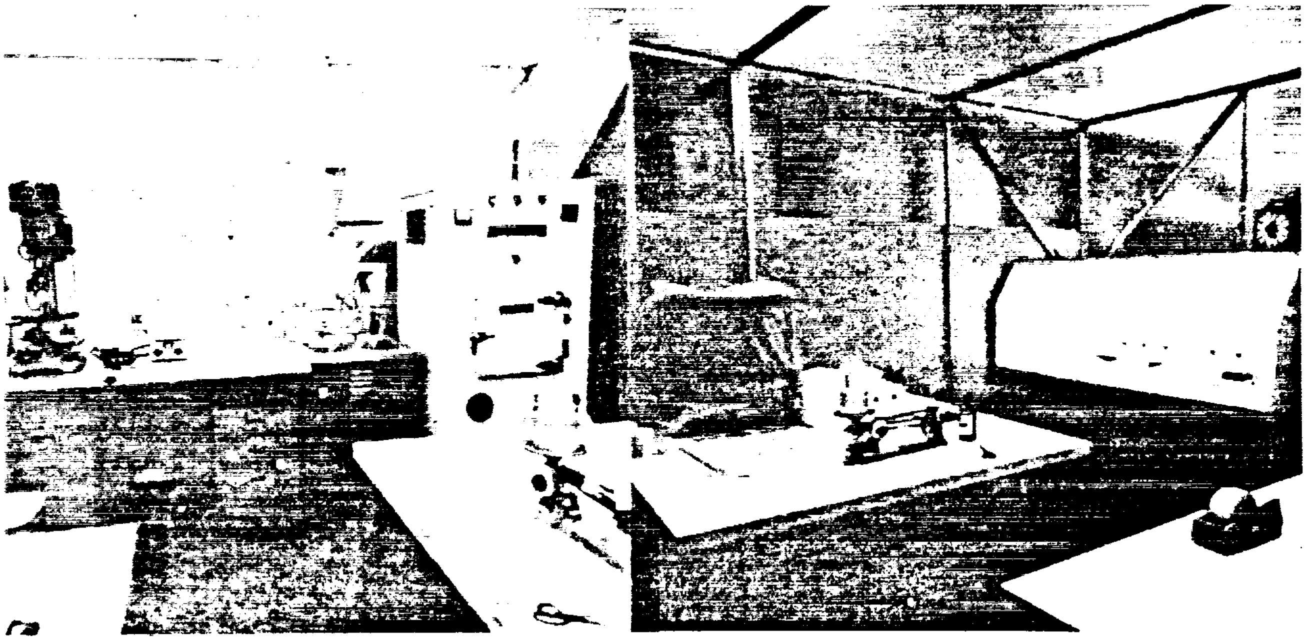


Figure 6-45. Purity Important to Amount of Energy Absorbed

is absorbed, for a given atmospheric entry for the three different purity levels of material. For example, the cumulative amount of energy absorbed up to about 5.5 ev, is approximately three percent for the hyperpure material, about twelve percent for the commercially pure slip cast material, and for the least pure material, about twenty-eight percent of the energy is abosrbed.

We have had some doubts, and people ask us, "How can you maintain this degree of purity?" It's really not that hard once you establish an area that you set aside and use only for this purpose. Figure 6-46 shows a room we put together with plastic film over some structure with normal laboratory equipment inside. There is no special equipment other than a few little items. For example, we can't let metallic materials come into contact with the SiO_2, so we coat metal components with plastic coatings.



CONTAINS: DUAL FILTERED, PRESSURIZED AIR
TEMPERATURE CONTROL (SEPARATED FROM CENTRAL AIRCONDITIONING)
LAMINAR FLOW BENCH
WET DIAMOND MACHINING FACILITY
MICROWAVE & AIR OVENS
SINK WITH DISTILLED WATER

Figure 6-46. High Purity Processing Room/Equipment

Other than that, just a normal, clean room environment. Again, we process only the very high purity SiO_2 material in this room.

In Figure 6-47 we will discuss a little about the morphology aspect which as you recall, has a large impact on reflectance. We have found that probably the most important processing variable which affects morphology is the degree of firing to which you subject the material. We want the reflectance to be as high as possible, and the density we want to be high for ablative reasons and strength reasons. What we have here is data for two different particle sizes of materials, both being hyperpure materials, made two different ways. The data at the left is for a material made by a normal ceramic process called dry processing. The data at the right is for a material made by the slip casting

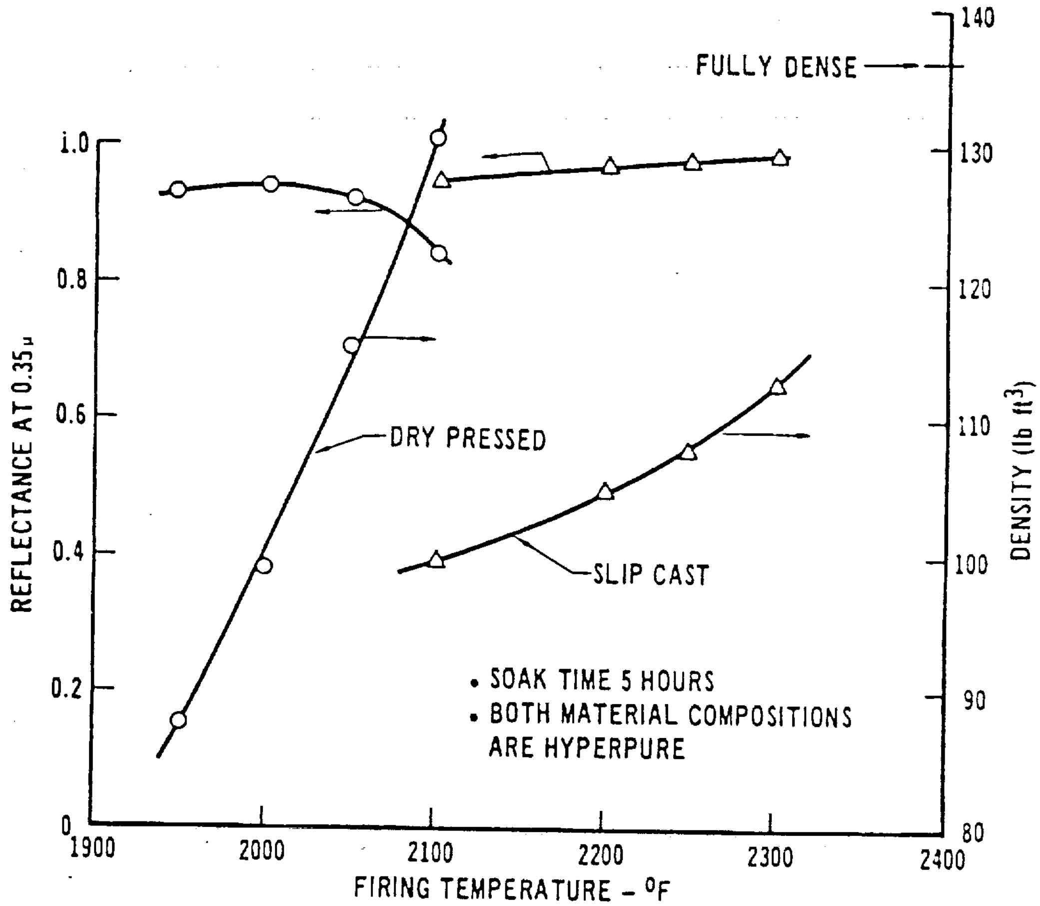


Figure 6-47. Reflectance and Density Change with Firing Temperature

process. We show data here for a dry pressed formulation containing a very small particle size, approximately .2 microns in diameter, silica as part of the charge. As we fire this material to higher temperatures, the density increases very rapidly and as it approaches the completely dense state, that is to say clear the reflectance begins to drop off. Plotted here is reflectance at 0.35 microns (we also have curves for other wavelengths). The slip case material has an average grain size of ten microns. The firing temperature has not yet been reached where we start to see a decrease in the reflectance at 0.35 microns. I think the proof of the material is in these two items reflectance and density.

Morphology can also be studied using the scanning electron microscope and we find this to be a very helpful tool, as Bill has discussed earlier. In Figure 6-48, the top row of pictures are 500x SEM's with firing temperature shown at the top of each picture. You can see a decrease in the size of the voids as temperature increases. The material is much smoother in texture as you proceed to the right. By viewing the same three specimens at approximately 10,000x (lower row) you can see the ultimate particles. As the firing temperature is increased, you can note a decrease in the angularity; the particles are becoming smoother. The sizes of the voids are diminishing.

In order to size these scattering type heat shields, we determine reflectance on a very thick sample and then a very thin sample, on the order of 0.750" and 0.020" respectively.

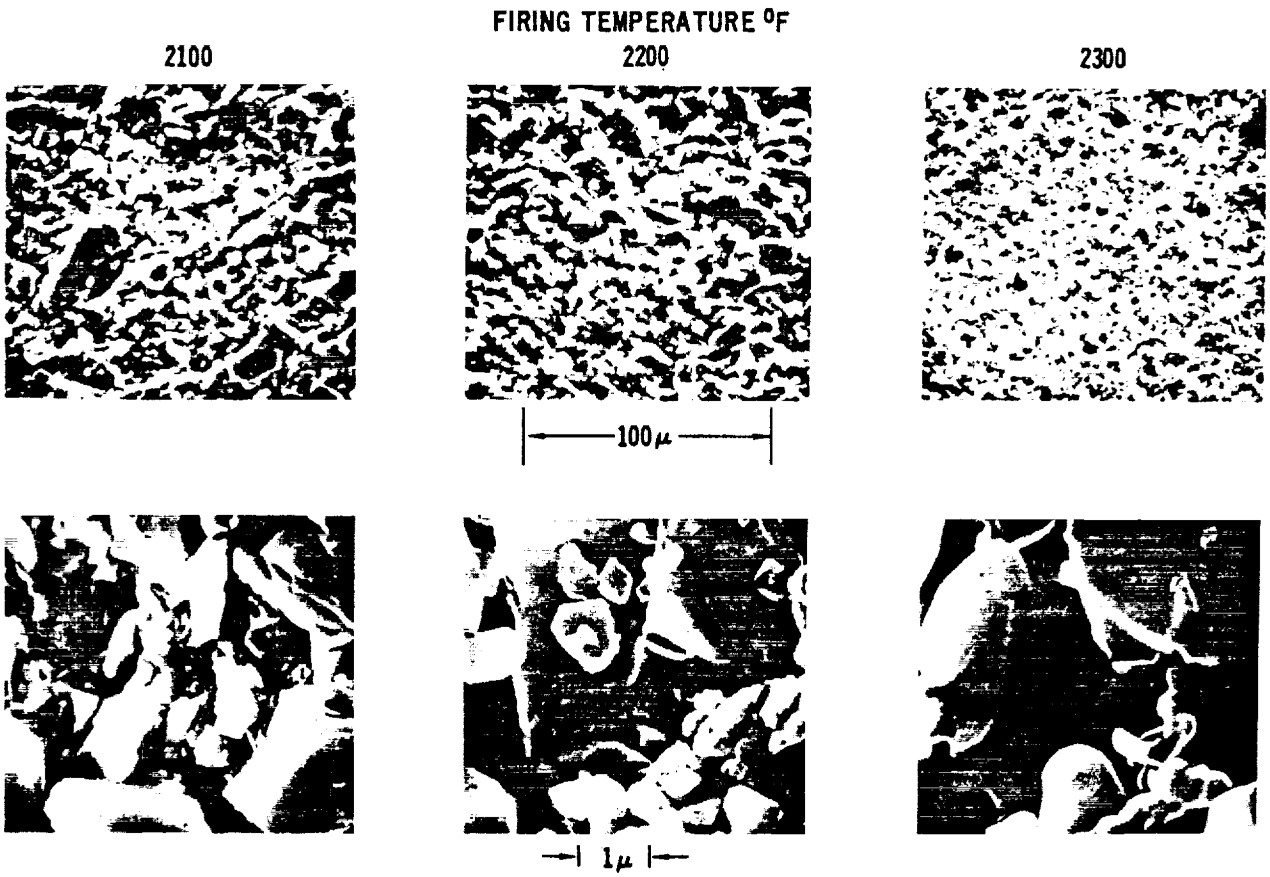


Figure 6-48. Morphology (Microstructure) Helps Explain Properties and Effects of Processing Variables.

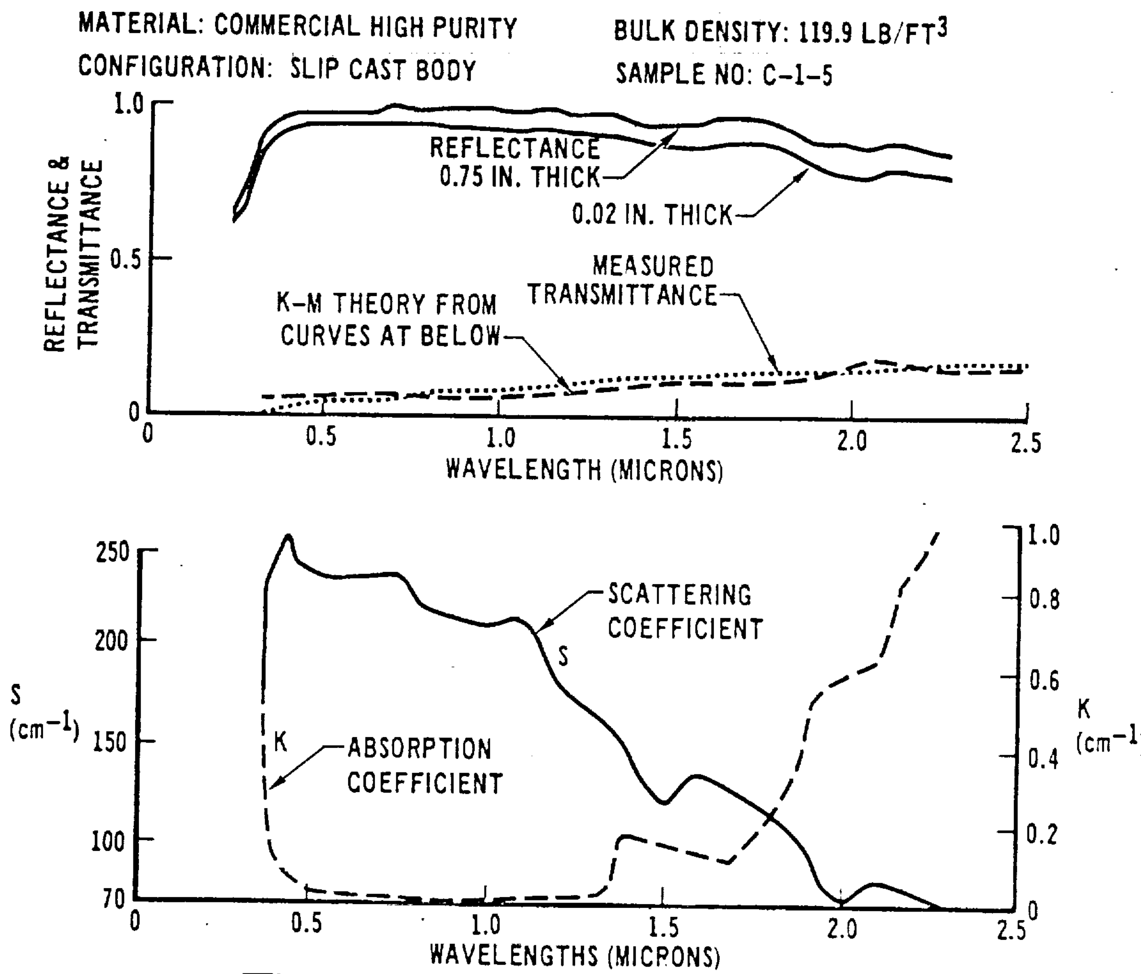


Figure 6-49. Kubelka-Munk Scattering Theory Used to Define Heat Shield Thickness

On the thin sample, we are getting some energy through. Then from that, we can calculate the scattering (S) and absorption (K) coefficients from reflectance data which then can be used in the computer program as John Howe has described. Typical data curves are shown in Figure 6-49.

Conclusions to date on our program are summarized in the table of Figure 6-50: purity and morphology are very important; that pure materials are available under one part per million from three suppliers; that required purity and morphology can be maintained. We feel that quite a high percentage of our steps in how to make this material are now understood. We have determined

- PURITY & MORPHOLOGY IMPORTANT FOR MAXIMUM REFLECTANCE
- PURE MATERIALS AVAILABLE ($\approx$1 PPM METALS)
- REQUIRED PURITY & MORPHOLOGY CAN BE MAINTAINED USING REASONABLE CARE
- 90% OF PROCESSING STEPS NOW DEFINED
- HIGH REFLECTANCE: 0.99 FROM 0.4 TO 1.2μ
0.90 FROM 0.24 TO 1.88μ
- READY TO BE SCALED UP TO FULL SIZED HEATSHIELDS
- READY TO CHARACTERIZE MATERIAL
- APPEARS TO BE COST EFFECTIVE

Figure 6-50. Conclusions

reflectance, 0.99 from 0.4 to 1.2 microns. We feel like our materials are developed to the point when we should talk about scaling up and producing samples of some size and should characterize the material, which we are doing now, in determining strength and stiffness. Cost appears to be in line with other heat shield materials.

UNIDENTIFIED SPEAKER: You speak of maintaining the purity. How far through the whole process of building this heat shield, putting it on the vehicle, having any number of mechanics and so on handling the thing all the way out to the salt water Cape, do you mean maintaining or do you mean achieving cleanliness in your environment?

MR. BLOME: Well, you obviously have to maintain purity. We found some real interesting things in this material. This high purity material opens up an entire new area of interest. You can take this material and fire it up to twenty-three or twenty-four hundred degrees Fahrenheit, and this is just not done now in the state-of-the-art. With other pure materials you start getting devitrification and things like that happening. Really, I think that if you can keep the purity internally or in other words if you can maintain a high purity inside the material, perhaps by sealing, by firing, or even packaging it can be maintained. It has to be done. You have to maintain the purity. We haven't taken any great pains, just the normal procedure in our R and D effort. We have made reasonably large sizes. This is a sample that we core drilled out some specimens for John Lundell at NASA Ames and this is the size that we have been able to make with good success.

DR. KLIORE: Looking at the plasma jet sample you passed around here, I notice some cracks in your surface.

MR. BLOME: That is in the glassy layer, yes.

DR. KLIORE: In connection with remarks made previously about good thermal shock resistance, do you have any comments on that?

MR. BLOME: I think those cracks that you see in the glass are from cool-down and from the contamination of the arc jet. It is a fact, we do get some contamination from the jet. That was exposed to a flux of about 3,600 $\frac{BTU}{ft^2\text{-sec}}$. So that specimen did have a good thermal shock load on it, and it did not come apart. Had we done that with an MgO or Al_2O_3 ceramic specimen, the pieces would be throughout the room, fractured from shock, I am sure. I have seen that happen.

QUESTION: How does the efficiency of the reflective heat shield compare to the black type? Let's say you encountered some warm atmosphere and you didn't have any radiation, or at least you had a low rate. Will it perform fairly comparable to the other type?

MR. BLOME: I think John Howe would be more competent to answer that then I would.

MR. HOWE: In a thoroughly convective environment, it doesn't perform as well as the carbon phenolic, that is, aside from the spallation effects, we don't really know. Silica has a very high sublimation energy, but it is only about half of that of carbon phenolic. So you would expect, in a purely convective environment, that you would need more silica than you would carbon phenolic.