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**FUNDAMENTAL MATERIALS RESEARCH**

**NUCLEATION INVESTIGATION AND CONTROL - THE NEXT DECADE AND**

**BEYOND IN SPACE MATERIALS AND SOFT MATTER RESEARCH**

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1. **Background**

Nucleation is the first step in almost all first-order phase transitions, which range from processes such as gas condensation to the crystallization of liquids. Nucleation occurs through the stochastic formation of small regions that are characteristic of the new phase [1]. The pervasiveness of nucleation makes it of significant fundamental interest, but it also has extreme practical importance. The ability to control nucleation is central for many important areas of materials science, including the refinement of solidified metallic alloys [2], glass formation [3, 4], and the production of glass ceramics [5]. Nucleation also plays an important role in the pharmaceutical [6, 7] and food [8, 9] industries, as well as in biological systems [10] and some medical diseases, such as the nucleation of pathogenic protein aggregates [11]. The nucleation step initiates the process that determines the properties and structure of the resulting product. Controlling nucleation is essential for almost all processing operations over a wide range of temperatures and types of materials. Successful development of space will require the ability to predict and control nucleation in materials used for future exploration and ultimately colonization. This white paper outlines key areas where research under different gravity conditions, ranging from Earth to Lunar to Mars to microgravity, can advance fundamental understanding of nucleation processes and lay the foundation for new materials and soft matter discovery and production. It is emphasized that a program of fundamental research in nucleation processes for different gravity condition is also relevant to several grand challenges that were identified by NASA in a recent soft matter workshop report (<https://ntrs.nasa.gov/citations/20205010493>).

 Broadly, there are two types of nucleation, homogeneous or heterogeneous [1], although recent research has blurred the distinction between these in some cases [12]. Homogeneous nucleation is the more fundamental type, occurring randomly in time and space within the initial phase. While still occurring randomly in time, heterogeneous nucleation occurs at specific catalytic sites such as surfaces or internal interfaces. Heterogeneous nucleation is more common and is indeed useful in processes such as grain refinement. Nucleation in a liquid is also typically heterogeneous, occurring at catalytic sites within the liquid (such as oxide particles) and on the wall of the container. If the container is removed (i.e. using containerless levitation methods), if the purity of the liquid is high, and if the studies are made under vacuum or in a high purity gas environment it is possible to study homogeneous nucleation. The microgravity environment is particularly useful for such studies, and it further allows the effect of stirring on nucleation to be examined.

 A central feature of both homogeneous and heterogenous nucleation is the existence of a nucleation barrier, which was first noted by Fahrenheit in his studies of the crystallization of water [13] and later by Turnbull in the crystallization of liquid metals [14]. Nucleation data are typically analyzed within the Classical Nucleation Theory (CNT), a phenomenological model developed over a century ago that incorporates the existence of the nucleation barrier [1]. It is possible in some cases to quantitatively fit nucleation data to the CNT, but the fit relies on adjustable parameters that cannot be independently checked, making it difficult, if not impossible, to make quantitative predictions. Also, there are serious problems with the thermodynamic aspects of the theory. Monte Carlo [15] and molecular dynamics [16] studies, as well as experimental studies of nucleation in colloidal systems [17] have clearly shown that a key assumption in CNT, i.e. a sharp interface between the nucleating cluster and the parent phase, is incorrect. Also, it is unlikely that macroscopic properties such as the Gibbs free energy and surface tension can accurately describe the thermodynamics of clusters containing only a few atoms, as is assumed in the CNT. Finally, since CNT was formulated for vapor condensation the importance of the structures of the parent liquid or glass phase are not considered. However, experimental studies have shown that in both metallic [12, 18] and silicate [19] liquids and glasses, structure can play a decisive role in crystal nucleation. Some of these points have been addressed by more advanced models, including density functional models (such as in [20]) and the phenomenological diffuse interface theory [21-23]. However, there exist only a very few cases where these advanced theories have been tested with experimental nucleation data [24, 25]. The kinetic model of CNT has fewer problems, but it cannot properly treat cases where the chemical compositions of the initial and final phases are different [26, 27], as has been demonstrated recently in the microgravity environment of the International Space Station.

1. **Recommendation and Benefits**

An increased level of study of nucleation processes is essential to test and extend the models discussed in the previous section. This is of course important for developing a deeper understanding of nucleation processes, but it also has significant practical implications. Some case studies that illustrate the measurement needs and their practical consequences are outlined in section 3. Some of the key points gleaned from these case studies are the need to:

1. Develop a deeper understanding of nucleation processes, including the effects of stirring and fluid flow that will have significant effects on space-based materials processing.
2. Improve the control of microstructural refinement during solidification and glass crystallization, enabling the microstructures to be tailored to specific applications.
3. Improve the understanding and control of additive manufacturing processes so that microstructural features that compromise the integrity of the manufactured components can be avoided.
4. Understand the effects of gravity on phase nucleation and growth, which will be needed for manufacturing in a variety of gravitational environments, such as in space on the Moon and on Mars.
5. Develop an improved ability to predict which systems will readily form glasses and the properties of those glasses.
6. Develop and validate advanced computer models for predicting nucleation behavior and solidification microstructures.
7. Measure dynamical and thermophysical properties of liquids and solidified phases.
8. Improve manufacturing techniques.

The US is facing a crisis, with a decreasing number of students seeking careers in science, technology, engineering, and math (STEM). The situation is even worse for women and minority students. An investment by NASA in the studies of the fundamentals of materials science and soft matter that are outlined will greatly enhance the training of the next generation of scientists, a benefit that cannot be overstated. In addition, space exploration will require the development of new skillsets obtained by performing research on samples in space.

1. **Examples of Relevant Nucleation Issues that Warrant Investigation**

**Nucleation during liquid solidification:** Quantitative measurements of homogeneous nucleation are necessary to test existing nucleation models and to guide the development of new models. By using containerless processing methods and making multiple measurements of the temperature where nucleation occurs in a supercooled liquid it is possible to deduce the thermodynamic and kinetic parameters [28]. The duration of experimental time on sounding rockets and parabolic flights is too short to obtain enough undercooling measurements; a platform that provide sustained periods of microgravity, such as on the International Space Station, is required. While of basic interest, these measurements are also technologically relevant. For example, although there has been a great deal of study into the growth of eutectic microstructures, there exist very few studies of how eutectics nucleate [28]. Since eutectic formation is a diffusion process, stirring must be significantly minimized to study this, which is only possible on a platform such as the ISS. Another example is grain refinement, i.e. obtaining a fine grain size, which is critically important for the mechanical properties of structural alloys. This is usually accomplished by the addition of grain refining agents that increase the heterogeneous nucleation rate, as is common in commercial aluminum alloys [29]. A quantitative understanding of how the grain refining agents work is still lacking, however.

**Fluid Motion Effects on Nucleation**: Liquids that are processed on Earth are always stirred by Marangoni and gravitationally induced flow, which can mask some features of nucleation, such as the influence of diffusion. Further, stirring is important for the crystallization of proteins [30] and pharmaceuticals [31]. The results of investigations of stirring effects in proteins and colloids are varied. Some indicate a decrease in the nucleation rate with increased stirring [32], while others have shown an increase in the nucleation rate [33, 34]. Some computer simulations have indicated the presence of a maximum in the nucleation rate as a function of stirring [35, 36]. More studies are needed to resolve these questions and to develop analytical models. Another effect of stirring that is relevant to materials processing is how it influence nucleation in cases where the crystallizing phase has a chemical composition that is different from that of the liquid (i.e., primary crystallization). Recent studies on the ISS have found that the nucleation rate is significantly increased with increased stirring. This is in agreement with a coupled flux model for nucleation [26] that connects the kinetics of diffusion with the attachment kinetics on the interface of nucleating clusters. Also, during rapid solidification with subsequent transformation from a metastable primary phase to a stable secondary phase, the time for the nucleation of the second phase is considerably shortened under the influence of convection, which significantly influences the microstructural evolution [37, 38]. Finally, there is evidence of cavitation induced nucleation during vigorous stirring [39]. Clearly, to gain control of the microstructure during solidification under varying gravitational environments further investigations are needed to identify the processes involved and to develop improved models for nucleation under these conditions. Since many biological processes that involve nucleation occur in the presence of stirring, these studies will also have an importance that extends beyond materials science.

**Additive Manufacturing:** Additive manufacturing, also known as 3-d printing, allows the construction of complex components through a layer-by-layer process. It is of significant commercial interest because it enables rapidly prototyping with reduced materials waste. It is of particular interest to NASA and the European Space Agency (ESA), since components that fail during a mission can be quickly manufactured without having to transport new ones from Earth, which would not be practical for bases on the Moon or Mars. A Moon Village has been proposed by ESA as the next step in the exploration of the Solar System, utilizing in-situ resources for its construction with additive manufacturing playing a central role [40]. These concepts have already been realized. As an example, a rocket engine pump that contains hundreds of different parts, including a turbine that spins at over 90,000 rpm, is shown in fig. 1. It was made entirely by additive manufacturing and has 45% fewer parts than one made using traditional manufacturing methods. During deep space exploration, additive manufacturing will be a key process ranging from habitat formation to on-demand manufacturing of essentials.

 

**Figure 1** – Rocket engine pump made by additive manufacturing (<https://www.nasa.gov/centers/marshall/news/news/releases/2015/successful-nasa-rocket-fuel-pump-tests-pave-way-for-3-d-printed-demonstrator-engine.html>).

 In one type of additive manufacturing new layers of the component are deposited by melting powders during repetitive scans using a highly concentrated heat source (often a laser). The challenge is to find the conditions that allow the microstructure of the component to be precisely determined, requiring control of the nucleation and growth in the solidifying layer. There are several different types of nucleation that must be considered in additive manufacturing (see figure 2 in [41]). These include homogeneous or heterogeneous nucleation in the melt pool, which is expected under the conditions of low thermal gradients in the liquid and high cooling rates, nucleation of the partially melted powders, non-epitaxial nucleation in the region next to the fusion boundary and epitaxial nucleation and growth on the partially melted grains on the fusion boundary. As in other types of solidification, it is important to be able to control the grain sizes, which can be enhanced by the addition of grain refining agents in the powder flow. The choice, size, and effectiveness of these refining agents, as well as the importance of stirring, need to be understood. Microstructural control, then, requires that the nucleation types are well understood and can be manipulated, that the thermal gradients are appropriate, and that stirring is correctly controlled. The variation of rheological properties with nucleation is also a key aspect for successful execution in varying gravity environments. All of this requires control of the addition of material, the heat input, the thermal loss, the strain/stress in the layers and the thermal history.

**Glass Formation and Glass Ceramics:** Glass formation is inherently determined by the avoidance of significant crystal nucleation and growth. The pre-factor for nucleation is assumed to reflect the diffusion coefficient in the liquid phase. However, since there have been very few measurements of the diffusion coefficient, the viscosity of the liquid is typically used to estimate this by assuming the Stokes-Einstein relation. The temperature dependence of the viscosity is characterized by a quantity called the fragility (i.e. strong or fragile), expressed in terms of an activation energy [42]. Strong liquids have a nearly constant activation energy from above the melting temperature to near the glass transition temperature (the temperature at which the supercooled liquid becomes a glass). The activation energy is small at high temperatures in fragile liquids but increases dramatically on approaching the glass transition temperature. Consequently, the magnitude of the viscosity for a strong liquid is larger than that of a fragile liquid in the supercooled region, which decreases the crystal nucleation rate and generally makes glass formation easier, although it is not universally true that a strong liquid will form a glass. Since the Stokes-Einstein relation is known to fail, even at relatively high temperatures, measurements of the diffusion coefficient are needed to accurately model crystal nucleation and hence glass formation. Additionally, recent studies suggest that the glass forming ability in metallic alloy liquids can be accurately predicted from measurements of the fragility and thermal expansion coefficient in the high temperature liquid [43]; whether this is true in other liquids is unclear. In either case, measurements of the dynamical and thermophysical properties in the liquid are needed, which can be obtained using levitation techniques in a micro-gravity environment. While silicate glasses tend to be brittle, partially crystallized glasses (glass ceramics) can be tough. Again, nucleation control is essential for obtaining the desired glass ceramic microstructures. Recent studies have shown that an apparent failure of CNT near the glass transition is instead an experimental artifact, suggesting that all previous measurements of the nucleation rate in this temperature range are flawed [44]. If it is to be possible to prepare glass ceramics from raw materials on the lunar surface and on Mars, an investment in fundamental nucleation studies in silicate glasses is critically needed.

**Measurements of Thermophysical Properties:** Quantitative modeling of microstructures requires accurate knowledge of thermophysical properties of the liquid and the crystallizing phases as a function of temperature. These include the liquid viscosity, surface tension, thermal conductivity, specific heats, thermal emissivity, electrical resistivity and enthalpy of transformation. Experimental studies on the ISS have shown that these quantities can be measured in a microgravity environment using electrostatic levitation and electromagnetic levitation techniques. These quantities can also provide new insight into processes such as potential liquid/liquid phase transitions and new fundamental physics. For example, the results of recent measurements of the electrical resistivity in metallic liquids as a function of temperature support MD predictions of a connection between liquid dynamics and suggest new physical behavior of electron localization at high temperatures [45].

**Computer Modeling:**  Computer modeling of microstructural development is playing an increasingly important role, with different numerical techniques used to probe processes at multiple length scales. Nucleation can be directly modeled at the microscopic scale by molecular dynamics and Monte Carlo methods. Nucleation and growth behavior as a function of stirring can be studied based on shear flow modeling calculations. Phase-field modeling and cellular automation simulations are important tools for predicting microstructural evolution at the mesoscale. Information that is needed of the equilibrium and nonequilibrium phase diagrams and the relative free energies of competing phases can be obtained from modeling methods such as CALPHAD. Investment in the continued development of numerical modeling approaches is critically needed to make the most of the experimental studies.

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