Introduction:

Glass-Ceramics (GC's) are critically relevant materials for industry and scientific research, primarily due to their outstanding mechanical, thermal, and optical properties. High-grade GC's such as lithium-aluminosilicate (LAS) are commonly used as insulation materials for high-performance aircrafts and missiles, optical bodies of precision optics, and biomaterials for medical equipment[1]. Although the material properties of GC's are very attractive in many engineering fields, the cost of manufacturing complex geometries can be prohibitive, primarily due to the high cost of tooling and limited machining capabilities of present manufacturing methods. Therefore, it is the goal of the proposed work to implement a novel method of manufacturing GC's with predictable, tailorable, and optimized material properties.

GC's are classified as two-phase materials; one being the glass matrix, the other being small volume fractions of nanocrystal inclusions. Typically, GC's are manufactured through casting or forming methods based on glass-making techniques. In these methods, the glass matrix is heated to high temperatures using a two-step process. In the first step, known as nucleation, the GC is heated just past its devitrification temperature to create a high density of nuclei throughout the interior of the glass. The second step involves re-heating the GC to a highly controllable temperature which directly impacts the growth rate, crystal size, and region of crystallization[2].

Vat Photopolymerization (VP) is an Additive Manufacturing (AM) process which utilizes UV light to selectively cure a polymer-based resin in a layer-by-layer fashion. VP can offer unparalleled resolution, complex internal and external features, and high-solid loadings of GC's to further enhance their applications. Digital Light Processing (DLP) is a sub-category of VP which uses a UV projector instead of a laser to expose cross-sections of the design geometry onto a resin vat. Therefore, the curing characteristics of the polymer resin are controlled by the light intensity and the effective pixel size from the projector. Additionally, by carefully tailoring the monomer, oligomer, and photo-initiator concentrations in the resin, high solid-loading of GC's within the resin could be achieved[3]. The polymer matrix is finally burnt off through a debinding step before the sintering process, resulting in a highly pure and fully dense GC part.

To further improve the mechanical, thermal, and optical properties of the GC's, an Ion-Exchange (IX) process will be implemented after the sintering step. During this process, cations of small atomic size from within the glass matrix surface are replaced by larger cations from the molten salt bath through a diffusion mechanism driven by temperature difference, resulting in a permanent compressive force in the surface of the part, which suppresses the growth of surface flaws and reduces crack propagation within the glass[4].

Proposed Research Activities:

Objective 1: Understanding primary and secondary parameter influence on material properties of Glass-Ceramics. From my previous and ongoing research, it has been noted that the concentrations of monomer, cross-linker, and photo-initiator in respect to the solid-loading of the matrix material greatly impact the printability and final properties of the GC. Therefore, it would be highly beneficial to understand the primary and secondary parameters during the printing, debinding, and sintering processes and their impact on the final material properties.

Primary parameters from the printing step could include UV exposure times, light intensity, and layer thickness; secondary printing parameters could include layer waiting time, lifting speed, and vat temperature. Degree of Conversion (DoC) is a common measurement tool that utilizes Fourier-Transform Infrared (FTIR) characterization data to rapidly quantify the progress of the photopolymerization reaction. Primary and secondary printing parameters will therefore be directly quantified and compared through DoC by use of FTIR. For debinding, primary parameters could include ramping rates and holding temperatures; secondary parameters could include crucible materials and furnace atmosphere. Initially, Thermogravimetric analysis (TGA) will be performed on printed samples to determine ideal holding temperatures in efforts to ensure complete removal of organic compounds. Additionally, Energy Dispersive Spectroscopy (EDS) will be used as an elemental analysis tool to compare anticipated composition of the GC's to actual results. In terms of sintering, primary parameters could include final sintering temperatures and furnace atmosphere; secondary parameters could include ramping rates and cooling rates. X-Ray Diffraction (XRD) analysis will be performed on sintered samples to broadly determine the degree of crystallization by comparing results to literature and standards. Finally, by identifying the relationships (linear or non-linear) between parameters and material properties, a novel, reliable, and well-understood manufacturing method for GC's based on the DLP process could be achieved.

Objective 2: Inclusion of alkali modifiers for chemical strengthening through Ion Exchange (IX). Based on the recent work of Gy, R.[4] and Macrelli, et al.[5], lithium, potassium, and sodium ion modifiers will be added to the GC resin formulation in preparation for chemical strengthening through the IX process, which will be carried out directly after Objective 1. The depth of penetration of the cationic exchange layer, also known as case depth, is a direct metric of the effectiveness of the IX process and will be evaluated at various depths using a refractometer. As mentioned before, the strengthening process intrinsically develops a residual compressive stress at the surface of the GC. Therefore, the effect of cation modifier concentration on the final mechanical properties will be assessed by compressive, flexural, and hardness testing based on ASTM standards and will be performed with equipment available at the Keck Center and SMP lab.

Extended Objective: Supporting the development of a machine-learning-based model to predict material properties of glasses from compositional data. Based on the recent work by Ward. et al[6], a framework capable of extracting predictive models from existing materials data is being developed by the Kansas City National Security Campus (KCNSC). My research will serve to provide the model with characterization and testing data obtained from Objectives 1 and 2 in order to effectively populate the model. *More detailed information may not be suitable for public release at this time.*

Intellectual Merit:

The proposed work represents a novel method for manufacturing two of the most relevant materials to society: ceramics and glasses. My research would directly advance the limited understanding of the intricate relationships between input parameters and material properties at different steps of the DLP manufacturing process. This critical understanding could potentially overcome a common barrier towards further development and implementation of DLP-based AM as a prevalent manufacturing method.

Broader Impact:

The development of DLP-based AM could enable previously unachievable part geometry of GC's and therefore, become a highly tailored process to impact many industries including aerospace, defense, and medical. Additionally, this work could drive further research in STEM, including fields such as Additive Manufacturing, Machine Learning, and materials science. Finally, the proposed work would directly support ongoing research efforts at the KCSNC, and thereby, the National Nuclear Security Administration.

References:

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