Machine learning-driven development of niobium-containing optical glasses

Desenvolvimento de vidros óticos contendo nióbio por machine learning

Dessarrollo de vidrios ópticos conteniendo nióbio mediante machine learning

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Abstract
High refractive index glasses are essential for old and new optical systems, such as microscopes, telescopes and novel augmented reality lenses and micro projectors. However, a fair portion of these glasses use toxic components, such as PbO, BaO, As2O3, and TeO2, which lead to high refractive indexes and facilitate the melting operation, but are harmful for human beings and the environment. On the other hand, it is known that niobium significantly increases the refractive index and is a non-toxic element. The objective of this paper was to develop new optical glass compositions containing Nb2O5 with a relatively high refractive index (n_d > 1.65), intermediate Abbe number (35 < V_d < 55) and fair glass transition temperature, T_g. To this end, we used a machine learning algorithm titled GLAS, which was recently developed at DEMA-UFSCar to produce new optical glasses composition. After running the algorithm 13 times, two of the most promising compositions were chosen and tested for their glass forming ability and other properties. The best composition was analyzed in respect to the refractive index, glass transition temperature and chemical durability. A comparison between the laboratory results and predictions of the artificial neural network indicates that the GLAS algorithm provides adequate formulations and can be immediately used for accelerating the design of new glasses, substantially reducing the laboratory testing effort. Also, the results indicate that niobium glasses might offer some advantages over its main competitor (La2O3).

Keywords: Optical glass; Niobium; Refractive index; Abbe number; Artificial intelligence; Machine learning.

Resumo
Vidros de alto índice de refração são essenciais para antigos e novos sistemas óticos, como microscópios, telescópios e novas lentes de realidade aumentada e microprojetores. No entanto, boa parte desses vidros utilizam componentes tóxicos, como PbO, BaO, As2O3 e TeO2, que levam a altos índices de refração e facilitam o processo de fusão, mas são prejudiciais ao ser humano e ao meio ambiente. Por outro lado, sabe-se que o nióbio aumenta significativamente o índice de refração e é um elemento que não é tóxico. O objetivo deste trabalho foi desenvolver novas composições de vidro ótico contendo Nb2O5 com índice de refração relativamente alto (n_d > 1.65), número Abbe intermediário (35 < V_d < 55) e temperatura de transição vítreo razoável, T_g. Para tanto, utilizamos um algoritmo de machine learning, GLAS, que foi desenvolvido recentemente no DEMA - UFSCar para produção de novas formulações de vidros óticos. Depois de rodar o algoritmo 13 vezes, duas das composições mais promissoras foram escolhidas e testadas por sua capacidade de formação de vidro e outras propriedades. A melhor composição foi analisada em relação ao índice de refração, temperatura de transição vítreo e durabilidade química. Uma comparação entre os resultados laboratoriais e as previsões da rede neural artificial indica que o algoritmo GLAS fornece formulações adequadas e pode ser usado imediatamente para acelerar o projeto de novos vidros, reduzindo substancialmente o número de testes laboratoriais. Além disso, os resultados indicam que os vidros de nióbio podem oferecer algumas vantagens sobre seu principal concorrente (La2O3).

Palavras-chave: Vidros óticos; Nióbio; Índice de refração; Número de abbe; Inteligência artificial; Machine learning.
Resumen
Vidrios de alto índice de refracción son esenciales para antiguos y nuevos sistemas ópticos, como los microscopios, los telescopios y las nuevas lentes de realidad aumentada y microproyectores. Sin embargo, la mayoría de estos vidrios utilizan componentes tóxicos, como PbO, BaO, As₂O₃ y TeO₂ que llevan a altos índices de refracción y facilitan el proceso de fusión, pero son perjudiciales para el ser humano y para el medio ambiente. Por otro lado, se sabe que el niobio aumenta significativamente el índice de refracción y es un elemento que no es tóxico. El objetivo de este trabajo fue desarrollar nuevas composiciones de vidrio óptico que contengan Nb₂O₅ con un índice de refracción relativamente alto (n_d > 1,65), un número Abbe intermediario (35 < V_d < 55) y una temperatura de transición vítrea razonable, Tg. Para ello, utilizamos un algoritmo de machine learning, GLAS, que fue desenvuelto recientemente en DEMa – UFSCar para la producción de nuevas formulaciones de vidrios ópticos. Tras ejecutar el algoritmo 13 veces, se eligieron dos de las composiciones más prometedoras y se comprobó su capacidad de formación de vidrio y otras propiedades. La mejor composición fue analizada con respecto al índice de refracción, la temperatura de transición vítrea y la durabilidad química. Una comparación entre los resultados de laboratorio y las predicciones de la red neural artificial muestra que el algoritmo GLAS proporciona formulaciones adecuadas y puede ser usado inmediatamente para acelerar el proyecto de nuevos vidrios, reduciendo sustancialmente el número de pruebas de laboratorio. Además de eso, los resultados indican que los vidrios de niobio pueden ofrecer algunas ventajas ante su principal competidor (La₂O₃).

Palabras clave: Vidrios ópticos; Niobio; Índice de refracción; Número de abbe; Inteligencia artificial; Machine learning.

1. Introduction

According Zanotto and Mauro (2017) natural glasses, such as obsidian and amber, have existed long before the emergence of life on earth. Synthetic oxide glasses were discovered circa 6,000 years ago and are now indispensable for domestic and high technology applications.

The term “optical glass” refers to high quality glass, chemically homogeneous, free of bubbles, inclusions and streaks, as shown in Figure 1.

![Figure 1 –Commercial Optical Glasses Schott and HOYA.](image)

Source: Schott (2021) and Hoya (2021).

Optical glasses are important players for leveraging the effect of optical systems for research and development activities and for many technologies used in the modern life (Hartmann et. al., 2010). However, a challenge in this segment is the scarcity of glasses having a combination of high refractive index (n_d) and high Abbe number (V_d) that, combined with other optical glass types, provide high resolution systems, i.e., low chromatic aberration with efficient color correction in small dimensions (fewer lenses, lighter weight) (Hartmann, et. al., 2010; Cassar, Santos and Zanotto, 2021). The research and development of optimized glass lenses has been focused on understanding the relationships between the chemical composition...
and properties, such as transmittance, refractive index, optical dispersion, and, in some cases, low glass transition temperature. Other important properties are the glass forming ability (GFA), that is the resistance against spontaneous crystallization during the manufacturing process, and the chemical durability. (Parsons, 1972; Hartmann et. al., 2010; Cassar, et al., 2021).

For the development of new optical systems, it is important to choose special glasses with ultra-high refractive index \( (n_d > 1.9) \). They are used in many applications, including AR/MR lenses and projectors (Cassar, et al., 2021; Parsons, 1972; Koudelka et. al., 2017). Unfortunately, some of these glasses contain toxic oxides, among them PbO, BaO, As\(_2\)O\(_3\), and TeO\(_2\), which lead to high refractive indexes and facilitate the fusion step (Bach, 1998; Sava et. al., 2009). The ROHS 2002/95/EC and WEEE 2002/96/EC directives, developed in 2002 and implemented in 2006 by the European Union, explicitly prohibit the use of lead in optical glass supplied as material for electronic devices (European Parliament, 2003). On the other hand, it is known that niobium and lanthanum significantly increase the refractive index and are non-toxic elements.

Niobium is abundant in nature with 4 oxidation states. The most stable is +5 (Nb\(_2\)O\(_5\)) as showed in Figure 02, which is called, niobium oxide. The niobium oxide is a substance extensively used in optical glasses to increase the refractive index (Koudelka et. al., 2017; Yasuma et. al., 2019; Xiangping et. al., 2020; Greenwood & Earnshaw, 1998) with the expense of decreasing its transmittance, due to Nb\(^{3+}\) and Nb\(^{4+}\) fractions that may appear during the melting stage (Chu et. al., 2011). Furthermore, studies carried out by Koudelka et. al., (2017), Chen et. al., (2012), Teixeira and Mazali, (2007) and Samuneva, Kralchev and Dimitrov (1991) indicate that glasses with high niobium oxide content have good chemical stability, which is highly desirable. (Figure 2)

**Figure 2 – Niobium Oxide.**

![Figure 2 – Niobium Oxide.](source: Niobium Tech (2021)).

Glass science and technology are currently experiencing the renaissance of “Artificial Intelligence”, due to the availability of powerful computational tools and hardware. The recent opening of access to the Sciglass database, which has approximately 400,000 entries on composition-properties provide fuel for data harvesting (Cassar, et al., 2018). Also, the INTERGLAD database provide valuable complementary information. Glasses have been so essential for the development of the modern civilization, that the UNESCO approved in 05/12/21 the year of 2022 as the International Year of Glass (http://www.iyog2022.org/)

In this work, we present a new optical glass composition with niobium, which was formulated by artificial intelligence (GLAS algorithm developed at DEMa-UFSCar) and the evidence of a successful laboratorial trial regarding the glass forming ability, refractive index, glass transition temperature, optical transmittance, and corrosion resistance. To compare the effect of
niobium oxide with its main competitor, lanthanum oxide, the same formulation was melted replacing the niobium oxide by lanthanum oxide with the same molar percentage, keeping the other components unchanged.

2. Methodology

For the development of this work, the formulation of optical glasses containing niobium were simulated by the machine learning algorithm denominated GLAS (Cassar, et al., 2021). Two series of glasses were investigated: silicate and phosphate glasses, both containing niobium oxide up to 5%wt. The simulation parameters were set to achieve compositions with a relatively high refractive index (n\(_q\) > 1.65), moderate Abbe number (V\(_p\) > 35) and low glass transition temperature.

2.1 Glass synthesis and analysis

After chosen the best compositions among those provided by the GLAS algorithm and, according to the desired parameters, some glasses were prepared by the melt-quenching technique. Commercial reagents of Calcium Carbonate (CaCO\(_3\), F.Maia 99.8%), Sodium Nitrate (NaNO\(_3\), BASE chemical 99.8%), Potassium Carbonate (K\(_2\)CO\(_3\), 99.8%) or Potassium Nitrate (KNO\(_3\), Neon, 99.8%), Antimony Trioxide (Sb\(_2\)O\(_3\), VETEC 99.8%), Silicon Oxide (sand, Jundu Mine 99.8% Fe < 50 ppm), Zetasil 99.8%, Fe< 10ppm), Ammonium Dihydrogen Phosphate (NH\(_4\)H\(_2\)PO\(_4\) F. Maia, 99.8%), Titanium Oxide (TiO\(_2\), Sigma Aldrich 99.9%), Magnesium Oxide (MgO, Synth 99.8%), Zinc Oxide (ZnO, SpeX, 99.8%), Tin Oxide (SnO\(_2\), Termofisher, 99.8%), Niobium Oxide (Nb\(_2\)O\(_5\), CBMM Optical Grade, 99.8%) or La\(_2\)O\(_3\) (Sigma Aldrich, 99.8%) and Strontium carbonate (SrCO\(_3\), Sigma Aldrich, 99.8%), or Strontium nitrate ((Sr(NO\(_3\))\(_2\) Sigma Aldrich (99.8%)) were previously dehydrated in muffle furnace during 12 hours at 100°C, or in the case of La\(_2\)O\(_3\) was calcined at 1000°C during 12 hours. The compositions chosen by the GLAS algorithm were weighted accordingly, mixed in stoichiometric amounts, and melted in a platinum crucible at 1200 -1500 °C in an electric furnace, in the presence of air. No crucible corrosion was observed. To improve the homogenization, the glass was remelted/crushed five times before the final casting. Finally, the pieces with size of order of 45 mm of length and thickness of 15 mm, were annealed at 50 °C below the calculated glass transition temperatures and slowly cooled down to room temperature. The bulk glass pieces were cut and optically polished before the characterization procedures. Furthermore, to compare the effect of Niobium with its main competitor, Lanthanum, the formulation with the best result was re-prepared by replacing the niobium oxide by lanthanum oxide, with the same molar percentage, keeping the other components.

The glass transition temperature was determined by differential scanning calorimetry (DSC) at a heating rate of 10 °C min\(^{-1}\) until 1000°C. The equipment used was a NETZSCH model DSC 404.

To investigate if any crystallization occurred during the cooling process, an X-ray diffraction analysis was performed. For this purpose, a Rigaku - Ultima IV diffraction meter with Bragg-Brentano configuration, and a Cu Kα radiation tube (1.54 Å) were used. The 20 angle was scanned from 20 to 80°, with a 0.02° step with an acquisition time of 3 seconds – “step-scan” mode. The samples were previously prepared by grinding using isopropyl alcohol and agate mortar. The phases were identified by using Crystallographica Search-Match Version 2.1.1.1 software, and indexed through the crystal structure database, BDEC – da CAPES, Inorganic Crystal Structure Database – ICSD. The refractive index was measured on polished samples with a Zeiss ABBE-PULFRICH refractometer.

The chemical durability of the bulk glasses was evaluated from the weight loss versus time of polished glass slabs immersed in 100 mL of 1.0 mol/L aqueous HCl solutions at 298 K in a polypropylene beaker. The measurements were made in duplicate. After each dissolution period, the slabs were removed from the beaker using polypropylene tweezers, dried on an absorbed paper and weighed. As the weight loss was approximately linear with the time, the chemical durability is expressed...
as the slope of the line in g/(cm² min). The surface area of the polished glass slabs was measured using a micrometer (Teixeira & Mazali, 2007). The formulation containing niobium oxide was compared with the commercial glass BK-7 (Schott).

3. Results and Discussion

3.1 Glass Formulations using the GLAS Algorithm and Laboratory Tests

During the search process, 13 compositions were suggested by the GLAS algorithm, all of which met the previously requested properties: refractive index (n_d > 1.65), moderate Abbe number (V_d > 35), and glass transition temperature (T_g < 850°C).

Using our accumulated knowledge on vitreous materials, the formulations 6 and 12 (Glass 6 and Glass12) were manually selected for testing some interesting properties, which included n_d, T_g, and the chemical durability (Table 1 and Table 2). Such knowledge allows for the choice of formulations with good GFA and other properties of interest, reducing the amount of trial-and-error testing as much as possible.

The produced glasses showed that the Nb_2O_5–phosphate glass (Glass 6), undergoes liquid phase separation, whereas the silicate glass with niobium - Glass 12 - shows a good glass forming ability (GFA), but presented a yellowish color. That may be due to the fact that the amount of nitrate in the batch mixture was not enough to maintain an oxidizing atmosphere to keep niobium in its most stable oxidation state (+5). Taking this fact into account, the Glass 12 was melted again with more nitrate sources, potassium and strontium carbonates (K_2CO_3 and SrCO_3) were replaced by their nitrate sources (KNO_3 and Sr(NO_3)_2) and the glass obtained was named Glass 14. A third test named Glass 16, was melted with the same formulation of Glass 14, which means increase the nitrate sources, but this time Mineração Jundu (level of purity ≥ 99,8% and Fe < 50ppm) sand was replaced by sand Zetasil 4 (level of purity ≥ 99,8% and Fe < 10ppm). 98%.

Table 1: Formulations and predicted properties suggested by the GLAS algorithm (Target), Phosphate Glass.

| Oxide   | Glass 6 (mol%) |
|---------|----------------|
| CaO     | 8,5            |
| K_2O    | 5,2            |
| Na_2O   |                |
| Sb_2O_3 | 1,5            |
| SiO_2   | 30,5           |
| P_2O_5  | 29,6           |
| TiO_2   | 0,9            |
| ZnO     | 0,9            |
| MgO     | 0,9            |
| SnO_2   | 1,5            |
| Nb_2O_5 | 14,3           |
| SrO     | 6,1            |
| n_d     | 1,70           |
| T_g (°C)| 686            |

Source: Authors.
Table 2: Formulations and predicted properties suggested by the GLAS algorithm (Target), Measured results: Silicate Glasses.

| Oxide | Glass 12 (mol%) | Glass 14 (mol%) | Glass 15 (mol%) | GLAS*1 (mol%) | Glass 16 (mol%) |
|-------|-----------------|-----------------|-----------------|--------------|----------------|
| CaO   | 4.9             | 4.9             | 4.9             | 4.44         | 4.9            |
| K₂O   | 12.7            | 12.7*           | 12.7*           | 12.59        | 12.7*          |
| Na₂O  | 2.4*            | 2.4*            | 2.4*            | 2.22         | 2.4*           |
| SiO₂  | 60.0            | 60.0            | 60.0            | 60           | 60.0           |
| ZnO   | 3.6             | 3.6             | 3.6             | 3.7          | 4.25           |
| MgO   | 2.4             | 2.4             | 2.4             | 2.22         | 2.42           |
| SnO₂  | 0.6             | 0.6             | 0.6             | 0.61         |                |
| Nb₂O₅ | 12.1            | 12.1            | 12.1            | 12.1         |                |
| La₂O₃ | 12.1            | 12.1            | 13.33           |              |                |
| SrO   | 1.2             | 1.2*            | 1.2*            | 1.48         | 1.2*           |

* Target | Measured

| T_g (°C) | Target | Measured |
|----------|--------|----------|
|          | 697    | 685      |
|          | 677    | 680      |

| n_a | Target | Measured |
|-----|--------|----------|
|     | 1.68   | 1.70 (+/- 0.02) |
|     | 1.63   | 1.70 (+/- 0.02) |

1 Properties suggested by the GLAS algorithm for a glass very similar to glass 15; *Nitrate source

Source: Authors.

As seen in Figure 3, Glasses 14 and 16 showed a good glass forming ability, but the strategy to avoid color (increased nitrate content – Glass 14 and increased nitrate content using Zetatsil instead of Mineração Jundu sand – Glass 16) had no effect and the formed glasses continued brown/yellowish.

**Figure 3:** Image Glass 14 and 16 formulation with niobium with good glass forming ability.

![Glass 14 and 16](image_url)
Glasses 14 and 16 were successfully produced. To compare the features of niobium oxide against its main competitor (Lanthanum oxide), the Glass 15 was melted by replacing Lanthanum by the same amount of niobium in molar %, keeping the other reagents unchanged (Table 2). To compare the Glass 15 target properties with measured results, the GLAS algorithm was used. It is important to emphasize, for the production of this silicate glass with 12.12% mol Nb₂O₅, 35.6% wt. niobium oxide is required. When this compound was replaced by lanthanum oxide, and due to its higher molecular weight, to obtain Glass 15 with the same molar mass, 40.4% wt. of lanthanum oxide was needed. Therefore, Glass 15 (La) has higher density than Glass 14 (Nb) and smaller refractive index indicating that lanthanum oxide is inferior to niobium oxide to achieve high refractive index.

It is relevant that when the % molar of niobium oxide was replaced by the same molar percentage of Lanthanum oxide, the glass (Glass 15) was dark, opaque and presented crystallization points. This result suggests that the Lanthanum modified composition is less resistant to crystallization that occurred on the cooling path.

The comparison between the target results from the GLAS algorithm and the measured results shown in Table 2, indicates that the target value provided by the GLAS program and the measured value are very similar, indicating the excellent GLAS predicting capacity. As for Glass 15, which is part vitrified and part crystallized, a comparison between the target T_g result was also very close, however, the refractive index value was not possible to be analyzed due to the lack of optical transparency.

As expected, the results of the thermal analysis of Glass 14 and 16 show inflexions characteristics of a glassy system representing a glass transition temperature. It appears at approximately 680°C and it is free of characteristic crystallization peaks indicating a good GFA. On the other hand, it is relevant to note that when the molar content of niobium oxide was replaced by the same molar% of lanthanum oxide, the glass obtained (Glass15) is dark and has some crystallization spots. In Figure 4, it is possible to verify that the Glass 15 with lanthanum has a glass transition temperature around 670°C, followed by a sharp exothermic peak, which indicates further crystallization at 800°C.
Figure 4: DSC analysis for the glasses 14 and 16 with niobium oxide and 15 with lanthanum oxide.

An X-ray diffraction analysis was also carried out on Glass 15 to certify the suspicion of crystal formation during the melting process. The results from figure 5 show the formation of crystalline structures of lanthanum silicates as well as a tin potassium silicate, proving that the Glass 15 presents poor glass forming ability. In this case, it is due to the Lanthanum content, which is the unique variable that has been changed when compared with Glass 14 which has shown good glass forming ability.
The corrosion resistance tests were performed through the loss of weight by surface area as a function of time of Glass 14 with niobium and Schott's commercial BK-7 lens when immersed in a 1mol/L HCl solution at room temperature. Figure 6 shows that the weight loss/(surface area.time) was approximately linear over time, indicating a constant rate of $6 \times 10^{-10}$ g (cm$^2$.min)$^{-1}$ for glass 14 and $2 \times 10^{-9}$ g (cm$^2$.min)$^{-1}$ for glass BK7 glass, after 27 days. So, the Glass 14, exhibits extremely low dissolution indicating that niobium might play an important role as a corrosion inhibitor.

According to Teixeira and Mazali (2007), their NAPT5 and NAPT1 phosphate glasses show extremely low dissolution (aqueous solution HCl 1mol/L), ranging from $6.2 \times 10^{-8}$ g(cm$^2$.min)$^{-1}$ to $1.8 \times 10^{-9}$ g(cm$^2$.min)$^{-1}$. NAPT5 glasses have a Nb/Ti ratio equal to 0.25, with a Nb$_2$O$_5$ content of 5% in mol%. On the other hand, NAPT1 glass has a Nb/Ti ratio equal to 4, with a Nb$_2$O$_5$ content of 30% in mol%. Therefore, the glass made with niobium is more resistant to corrosion than the researched phosphate glasses.
4. Conclusion

A fertile field for machine learning-driven development exists for efficient new glass competitions. In this paperwork, a machine learning algorithm is presented as a successfully alternative used to design new optical glass compositions with positive combinations of refractive index and glass transition temperature, with such as glasses 12 and 14, which also show good glass forming ability. The target property values provided by the GLAS algorithm are very similar to the experimental data indicating that this algorithm can be used directly for new glass formulation design, leveraging the development of new optical glasses.

Despite our long experience with empirical glass formulations, it was not possible to predict the presence of LLPS in the phosphate glass (Glass 6), confirming the need for ML strategies working together with improved engineering knowledge for eliminating unnecessary laboratory tests.

Overall it can be concluded that: (i) niobium oxide is not hygroscopic, it can be used as is, without the need of heat treatment before use. In contrast, its main competitor, lanthanum oxide is hygroscopic at room temperature, requiring calcination at 1000°C for 12 hours; (ii) When the molar percentage of niobium oxide was replaced by lanthanum oxide in Glass 15, the resulting glass was dark and partially crystallized. (iii) Simulations via the GLAS algorithm indicated that niobium oxide has a greater power to increase the refractive index than lanthanum oxide for the same molar mass; (iv) The Glass 14 exhibits very low dissolution in HCl 1mol/L aqueous solution indicating that niobium maybe plays an important role as a corrosion inhibitor in glasses. The corrosion test results have shown a corrosion inhibition rate constant of $6 \times 10^{-10}$ g(cm$^2$min$^{-1}$) for the silicate glass with niobium (Glass 14) and $2 \times 10^{-9}$ g(cm$^2$min$^{-1}$) for the BK7 glass, after 27 days. (5) The increase of nitrate in new glass compositions with niobium were not enough to obtain colorless glass, so it will be necessary to find new strategies to obtain high transmittance, to obtain a colorless niobium silicate glass.

For the continuation of this work, we would like: (i) To try to find compositions with approximately the same properties that we already found for “Nb$_2$O$_5$-based” glass samples, with an even lower number of components, to simplify a possible industrial scale fabrication; (ii) Comparison of the Nb$_2$O$_5$ properties found for these glassy compositions with others having similar mol% oxides, for example, ZrO$_2$ and ZnO; (iii) To try to find via ML “Nb$_2$O$_5$-based” glass compositions with even higher $n_d > 1.7$ which could to be applicable to lenses with lower spherical aberration and miniaturized dimensions, and (iv) ML “Nb$_2$O$_5$-based” glass compositions with higher $V_d > 60$, suitable to optical systems with lower chromatic dispersion.
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