**Title and Subtitle**

Novel Processing of Infrared Transmitting ZrO2-ZrW2O8 Nanocomposites

**Abstract**

In our research we were able to synthesize ZrW2O8 and Zr(W,Mo)2O8 single crystals to determine some basic properties including IR transparency as a function of temperature, hardness, and elastic modulus. We were also successful in preparing nanopowders with varying Mo content and determined structure, morphology, CTE, thermal stability as well as infrared transparency. Compatibility of Zr(W,Mo)2O8 with ZrO2 has also been established while other oxides reacted with Zr(W,Mo)2O8. Attempts to process fully dense nanocomposite by traditional sintering were not successful. Highest density we were able achieve was 92%. Densification of ZrW2O8 is limited by its thermodynamic stability (1378-1530 K) and high volatility of (W,Mo). Therefore, novel processing approaches such as melt spinning and flash sintering. The latter seemed to produce nanostructured nearly fully dense nanocomposites. ZrO2 grains were uniformly distributed in the matrix of ZrW2O8. However, the process optimization, IR transmittance and mechanical properties of the nanocomposites were not completed due to expiration of the grant.

**Subject Terms**

ZrW2O8, Nanocomposite, IR transmittance, Negative CTE.
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Novel Processing of Infrared Transmitting ZrO2-ZrW2O8 Nanocomposites
(Award Number: N00014-12-1-0316)

Final Report

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September 2013
Synopsis
This is a final technical report on grant # N00014-12-1-0316 entitled “Novel Processing of Infrared Transmitting ZrO₂-ZrW₂O₈ Nanocomposites” which was a six-month extension of a previous grant. Hence it only covers six months of research activity. During this period, several non-traditional processing approaches were attempted to obtain dense nanocomposites. Previous grant attempted to process Y₂O₃/ZrW₂O₈ composite. High affinity of Y₂O₃ to react with ZrW₂O₈ made this composite unattainable. As a result we proposed to investigate ZrC-ZrW₂O₈ nanocomposite as ZrO₂ coexists over a large composition range as shown in the binary phase diagram.

To achieve the stated objective, the following tasks were undertaken:
• Compact and sinter ZrW₂O₈ and Zr(W,Mo)₂O₈ to achieve full density. Characterize dense pellets for IR transparency and CTE
• Process Zr(W,Mo)₂O₈-ZrO₂ nanocomposites to obtain dense pellets

Technical Approach
Performance requirements for the IR window and domes for high speed missiles are challenging and a non-trivial problem. The challenges stem not only due to the lack of basic data on the individual components but also from the difficulty of processing to achieve fully dense, high purity, and strong composite material that transmits in the mid IR range.

The properties of the nanocomposite would not only depend on its constituents but also on other factors such as residual porosity and grain size. The choice of sintering temperature, in this case, is dictated by the high temperature stability of Zr(W,Mo)₂O₈ compound. The sintering has to be carried out within the stable temperature range (1105< T <1257°C) of Zr(W,Mo)₂O₈. Therefore, sintering behavior and IR transmittance of nanocrystalline Zr(W,Mo)₂O₈ compacts need to be established.

In previous grant period, we synthesized nano powders and single crystals of ZrW₂O₈ and Zr(W,Mo)₂O₈ and characterize them for microstructure, temperature stability and CTE

Summary
Single crystals of ZrW₂O₈ having sizes up to 4 mm were synthesized. The Laue’s diffraction pattern confirmed the single crystallographic domain of the grown crystals. Room temperature FTIR showed that the crystals were transparent in the mid-IR regime. The crystals were characterized for mechanical properties and IR transmittance as a function of temperature.

Single crystals of ZrMo₂O₈ were successfully grown using Li₂MoO₄ as flux. To the best of our knowledge, this is the first time growth of ZrMo₂O₈ single crystal was demonstrated. Single crystal XRD refinement showed that the grown crystals had the monoclinic crystal structure. Laue diffraction confirmed that 1-3 mm crystal samples were actually single crystals. The mechanical properties of ZrMo₂O₈ were also investigated using nanoindentation method.

Nanocrystalline ZrW₂₋ₓMoₓO₈ with varying δ (0 ≤ δ ≤ 1) was synthesized by the hydrothermal route. It was found that the increasing amounts of Mo substitution suppress the α→β transformation
temperature eliminating the discontinuity in the thermal expansion coefficient in the potential application temperature range. High temperature stability of nanocrystalline ZrW$_2$MoO$_8$ powders was studied. Substitution of Mo raises the decomposition temperature of zirconium tungstate up to 850 °C at which Mo evaporation in the form of MoO$_3$ destabilizes the structure. Molybdenum substitution essentially increases the high temperature stability of ZrW$_2$O$_8$ by about 100 °C.

Densification behavior of hydrothermally synthesized ZrW$_2$O$_8$ nanopowders was also studied. Compacts with 90% theoretical density was achieved by ambient pressure sintering. The sintering temperature regime was dictated by the narrow stability temperature range of ZrW$_2$O$_8$ (1105-1257 °C) while densification in that temperature regime was found to be limited by the evaporation of WO$_3$. Due to these reasons achieving a fully dense composite based on ZrW$_2$O$_8$ pose a formidable scientific challenge. Several novel processing approaches were attempted to overcome this challenge.

Also, we noted slow hydration of ZrW$_2$O$_8$ under ambient conditions might be concern especially for the powders. We investigated effect of hydration on the negative coefficient of thermal expansion (NCTE) of ZrW$_2$O$_8$. On storing under ambient conditions for the 6-month, about 66% of the outer annular volume was hydrated to ZrW$_2$O$_8\cdot0.35$H$_2$O while 1-year sample stored was hydrated to ZrW$_2$O$_8\cdot0.72$H$_2$O. NCTE was characterized by in-situ high temperature X-ray diffraction (XRD) measurements between 25-200 °C. XPS and TGA were used to characterize the nature of bonding of water molecules in the ZrW$_2$O$_8$ structure. The NCTE of ZrW$_2$O$_8\cdot0.35$H$_2$O remained intact while on further hydration to ZrW$_2$O$_8\cdot0.72$H$_2$O the negative CTE was lost. The water molecules were bonded stronger than adsorbed water molecules while weaker than of hydroxyl ions.

Progress: September 2012- March 2013

Sintering studies of ZrO$_2$-ZrW$_2$O$_8$ compacts

Processing of infrared transparent nanocomposite with near zero CTE by mixing negative and positive CTE oxides was originally proposed. We have shown that Zr(W,Mo)O$_8$ has a negative CTE over a wide range of temperature (up to 998 K). The positive CTE of ZrO$_2$ (9.6x10^{-6} K$^{-1}$) is also comparable to that of the negative CTE value of Zr(W,Mo)$_2$O$_8$ (~ -5x10^{-6} K$^{-1}$). Traditional sintering does not yield fully dense and nanostructured material due to high volatility of the (W,Mo)O$_3$ and long sintering times. The nanostructured powder compacts are densified employing recently developed “flash sintering” technique.

Accomplishments
In our research we were able to synthesize ZrW$_2$O$_8$ and Zr(W,Mo)$_2$O$_8$ single crystals to determine some basic properties including IR transparency as a function of temperature, hardness, and elastic modulus. We were also successful in preparing nanopowders with varying Mo content and determined structure, morphology, CTE, thermal stability as well as infrared transparency. Compatibility of Zr(W,Mo)$_2$O$_8$ with ZrO$_2$ has also been established while other oxides reacted with Zr(W,Mo)$_2$O$_8$. Attempts to process fully dense nanocomposite by traditional sintering were not successful. Highest density we were able achieve was 92%. Densification of ZrW$_2$O$_8$ is limited by its thermodynamic stability (1378-1530 K) and high volatility of (W,Mo). Therefore, a very fast sintering process or low temperature densification needs to be employed.

Flash sintering
Flash sintering is a novel sintering route recently developed by Raj and coworkers where sintering is assisted by application of a DC electrical field to the specimen under ambient pressure. A characteristic feature of this process is sudden densification above a threshold electric field. This phenomenon has been demonstrated for several oxides such as zirconia, magnesia doped alumina, magnesium-aluminate spinel etc. The short sintering time and ambient atmosphere is ideal for densification of ZrW$_2$O$_8$. Therefore, we believe that this process is very promising for achieving close to 100% densification in ZrW$_2$O$_8$-ZrO$_2$ nanocomposites. In fact, a few preliminary studies with a crude setup we put together recently gave very promising results as shown in Figure 1.
Preliminary results
In order to explore the feasibility of flash sintering route, we carried out preliminary experiments in our lab using a manual power supply and a tube furnace. Promising densification was observed. Flash sintered samples show much denser microstructures compared to conventionally sintered samples. SEM images of conventionally sintered and flash sintered samples are depicted in Figure 1.

![SEM images of conventionally sintered and flash sintered samples](image)

Figure 1. SEM image of ZrW$_2$O$_8$-ZrO$_2$ (2:1 wt. ratio) composites a) conventionally sintered at 1200 °C for 30 min b) flash sintered at 1150 °C for 3 min.

**Densification behavior of ZrW$_2$O$_8$**
Limited literature is available on the densification behavior of ZrW$_2$O$_8$ and its compatibility with various oxides. We have studied the sintering behavior of hydrothermally synthesized ZrW$_2$O$_8$ nanopowders. The pellets were preheated at 600 °C for 30 minutes before transferring to preheated sintering furnace at temperatures within the stability regime and times from 5 min to 2 h. So far, density up to a maximum of about 92% (as estimated from Archimedes' principle) was achieved at 1175 °C for 1 h. SEM micrograph of the cross section of such a pellet is shown in Figure 2. Longer holding times resulted in evaporation of WO$_3$ consequently the reduction in density and formation of ZrO$_2$. Therefore, it was thought that the enhanced diffusivity at increased temperatures could help in closing the residual pores. However, increasing the sintering temperature to 1200 °C resulted in complete melting of the pellet. This is an unusual behavior as the melting temperature of ZrW$_2$O$_8$ is 1257°C. The melting might be due to the presence of residual sodium which comes from the added salt during hydrothermal synthesis of ZrW$_2$O$_8$ nanopowders. At the same time the existence of non-stoichiometric compounds at grain boundaries should not be ruled out. In the present objectives and constraints of the current study, a detailed analysis of this lower-than-expected melting temperature behavior was not studied in detail.

**ZrW$_2$O$_8$-ZrO$_2$ composites**
Several approaches were explored for the synthesis of ZrW$_2$O$_8$-ZrO$_2$ nanocomposites.

**Ball Milling:** In this approach, a mixture of ZrO$_2$ and WO$_3$ in appropriate ratio was ball milled for varying times which were subsequently pelletized in a uniaxial press. The pellets were sintered in the
temperature range between 1125-1210 °C for varying sintering times of 5 min to 2 h followed by mild quenching.

**Powder mixing:** In this approach, hydrothermally synthesized nanocrystalline ZrW$_2$O$_8$ and commercially purchased ZrO$_2$ nanopowders were thoroughly mixed in appropriate ratio. The powder mixture was pelletized and sintered. In order to avoid any decomposition of ZrW$_2$O$_8$, the pellets were preheated to about 600 °C before transferring to sintering furnace at 1150 °C.

In both cases, evaporation of WO$_3$ from outer layers of pellets was observed at higher sintering times and temperatures. A sintered density up to about 92% is achieved on sintering for upto 30 min at 1200 °C. However, the exact ZrW$_2$O$_8$:ZrO$_2$ ratio and composition gradient in the sintered pellets need to be ascertained.

**Sintering in a sealed glass tube:** In order to minimize the loss of WO$_3$, the powder mixture of WO$_3$ and ZrO$_2$ was cold isostatically pressed and sealed in a pyrex glass capsules before heating to about 1175 °C. If successful this approach was thought to be used for pressure assisted sintering as this will not only save the container of the hot isostatic press from reacting with WO$_3$ but less than 90% dense pellets could also be densified. However, the pyrex glass softened and penetrated through the cold isostatically pressed samples. While, other high temperature glasses (Vycor or Quartz) would not be soft enough at the planned sintering temperature regime to transfer the pressure without shattering during hot isostatic pressing.

**Hydrothermal synthesis of ZrW$_2$O$_8$-ZrO$_2$ powder mixture:** Up to this point, the processing approach has been to synthesize ZrW$_2$O$_8$-ZrO$_2$ nanocomposite by mechanical mixing and pelletizing the hydrothermally synthesized nanocrystalline ZrW$_2$O$_8$ powders with the commercially available nanocrystalline ZrO$_2$ before sintering at around 1150-1200 °C. This was being done with the expectation that the ZrO$_2$, having a much higher melting point (~2750 °C) compared to that of ZrW$_2$O$_8$, would restrict the grain growth of ZrW$_2$O$_8$. However, the SEM images of the polished sections of sintered pellets showed regions of large areas of ZrW$_2$O$_8$ along with well grown ZrO$_2$ particles which indicated to the fact that the mechanical mixing was ineffective in preventing agglomeration of particle and achieving a uniform distribution of ZrO$_2$ and ZrW$_2$O$_8$. To achieve homogeneous mixture of the components at the nano scale, appropriate amounts of ZrO$_2$ nanopowder was added to the starting solution prior to hydrothermal synthesis of nanocrystalline ZrW$_2$O$_8$.

This approach resulted in much better distribution of ZrO$_2$ in the matrix of ZrW$_2$O$_8$. Comparison of XRD patterns of pure ZrW$_2$O$_8$ to that of the composite indicates that indeed we achieved a composite microstructure with ZrO$_2$ and ZrW$_2$O$_8$. No other phase or compound was detected (See Figure 4a). However, some of the ZrO$_2$ particles were of the order of several microns (Obviously, nanoscale ZrO$_2$ particles were not discernible at this magnification). This observation indicated the presence of clusters of ZrO$_2$ nanoparticles which on heating to sintering temperatures grew rapidly as can be seen in the
SEM image depicted in Figure 4b. This phenomenon was observed in all three samples synthesized with ZrW$_2$O$_8$/ZrO$_2$ mass ratios of 1, 2 and 4. This shows that the problem was due to the presence of clusters in ZrO$_2$. Different processing approaches are being explored to obtain finely distributed ZrO$_2$ nanoparticles in the final composite.

![Figure 4 a) XRD of hydrothermally synthesized pure and composite powder, b) SEM (BSE) image of composite from hydrothermally synthesized composite powders shows good distribution of ZrO$_2$ but appreciable grains (sintered at 1200 °C for 30 min)](image)

**CTE of composites**

CTE of the synthesized composites prepared by sintering the hydrothermally synthesized ZrO$_2$-ZrW$_2$O$_8$ composite powders were measured using a thermo-mechanical analyzer (TMA). Figure 5 depicts the change in dimension as a function of temperature for composites having varying ZrO$_2$ fractions. CTE of the composites progressively increased and approached zero with increasing ZrO$_2$ volume fractions. Lowest CTE of about -1.63 ppm was achieved for the composite having 0.44 volume fraction of ZrO$_2$ as can be observed from Table 2. Composites sintered at 1175 °C showed lower CTE when compared with the composites sintered at 1150 °C as expected.

![Figure 5. Change in length as a function of temperature for ZrW$_2$O$_8$-ZrO$_2$ composites having varying ZrO$_2$ volumes](image)

<table>
<thead>
<tr>
<th>Volume fraction ZrO$_2$</th>
<th>CTE (30-100 °C) ppm</th>
<th>CTE (200-500) ppm</th>
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<tbody>
<tr>
<td>0</td>
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<tr>
<td>0.14</td>
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<tr>
<td>0.44</td>
<td>-2.8</td>
<td>-1.63</td>
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<table>
<thead>
<tr>
<th>Sintering Temperature</th>
<th>CTE (30-100 °C) ppm</th>
<th>CTE (200-500) ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1150</td>
<td>-2.8</td>
<td>-1.63</td>
</tr>
<tr>
<td>1175</td>
<td>-3.34</td>
<td>-2.3</td>
</tr>
</tbody>
</table>

Table 2. CTE of ZrW$_2$O$_8$:ZrO$_2$ composite having varying ZrO$_2$ volume fractions

Table 2a. CTE of ZrW$_2$O$_8$:ZrO$_2$ (1:0.78) composite sintered at different temperature


**International refereed journals**


2. Md. Imteyaz Ahmad, Kate Lindley and Mufit Akinc, “Hydrothermal synthesis of ZrW$_{2.5}$Mo$_3$O$_8$ (δ=0-0.91) and its α→β transformation”, *J. Am. Ceram. Soc.*, 94 (8) (2011) 2619-2624


**Conference Presentations**

1. Md. Imteyaz Ahmad and Mufit Akinc, Effect of Mo substitution on the phase transformation of ZrW$_2$O$_8$, 39th Annual Conference of the North American Thermal Analysis Society (NATAS), August 8-10, 2011, Des Moines, Iowa, USA

2. Md. Imteyaz Ahmad and Mufit Akinc, Negative Thermal Expansion ZrW$_2$O$_8$: Powders and Single Crystals, 7th International Conference on High Performance Ceramics, November 4-7, 2011, Xiamen, China


4. Md. Imteyaz Ahmad and Mufit Akinc, CTE tailored ZrW$_2$O$_8$-ZrO$_2$ nanocomposite, 36th International Conference & Exposition on Advanced Ceramics & Composites (ICACC), January 22-27, 2012, Daytona Beach, Florida

**References**


4. Li Sun and Patrick Kwon, "ZrW\textsubscript{2}O\textsubscript{8}/ZrO\textsubscript{2} composites by in situ synthesis of ZrO\textsubscript{2}+WO\textsubscript{3}; Processing, coefficient of thermal expansion, and theoretical model prediction", Mat. Sci. Engg. A, 527 (2009) 93


6. Li Sun and Patrick Kwon, "ZrW\textsubscript{2}O\textsubscript{8}–ZrO\textsubscript{2} Continuous Functionally Graded Materials Fabricated by In Situ Reaction of ZrO\textsubscript{2} and WO\textsubscript{3}", J. Am. Ceram. Soc., 93 [3] (2010) 703
for '||' "CNWDI document. Primary code must be 4, or 5.';
out_status := -1;
END IF;
END IF;

-- Check for NOFORN documents
THEN
IF ( (INSTR(special_indicator_text, web_global_var_pkg.NOFORN)) > 0 ) THEN

IF (( distrib_codel != web_global_var_pkg.DISTRIB_02 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_03 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_04 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_04_34 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_05 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_16 )) THEN

special_indicator_error := 'Wrong Primary Distribution ' ||
'code for NOFORN document. Primary code must be ' ||
'2, 3, 4, 16 or 5.';
distribution_error := 'Wrong Primary Distribution code
for '||'
'NOFORN document. Primary code must be 2, 3, 4, 16
or 5.';
out_status := -1;
END IF;

-- Check for REL_TO documents
THEN
IF ( (INSTR(special_indicator_text, web_global_var_pkg.REL_TO)) > 0 ) THEN

IF (( distrib_codel != web_global_var_pkg.DISTRIB_02 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_03 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_04 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_04_34 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_05 )
AND ( distrib_codel != web_global_var_pkg.DISTRIB_16 )) THEN

special_indicator_error := 'Wrong Primary Distribution ' ||
'code for REL_TO document. Primary code must be ' ||
'2, 3, 4, 16 or 5.';
distribution_error := 'Wrong Primary Distribution code
for '||'
'REL_TO document. Primary code must be 2, 3, 4, 16
or 5.';
out_status := -1;
END IF;

END IF;

END IF;

-- EXCEPTION
WHEN OTHERS THEN
errors_pkg.dump(SQLCODE, SQLERRM, loc_procedure_name);
out_status := SQLCODE;
PROCEDURE doval_citation_classification
(
abstract_class IN NUMBER, --field_28
citation_class IN NUMBER, --field_03
document_class IN NUMBER, --field_20
identifiers_class IN NUMBER, --field_26
title_class IN NUMBER, --field_08
abstract_class_error OUT VARCHAR2,
citation_class_error OUT VARCHAR2,
document_class_error OUT VARCHAR2,
identifiers_class_error OUT VARCHAR2,
title_class_error OUT VARCHAR2,
out_status OUT NUMBER
) IS

loc_doc_class := document_class;
loc_procedure_name VARCHAR2(100) :=

'web_validation_pkg.doval_citation_classification';

BEGIN

abstract_class_error := '';
citation_class_error := '';
document_class_error := '';
identifiers_class_error := '';
title_class_error := '';
out_status := 0;

-- Classification are 1-4 for non-nato and 5-8 for nato
-- This adjusts the class down to just 1-4 so I don't have to
-- keep making double checks
IF ( loc_doc_class >= web_global_var_pkg.CLASS_UNCLASSIFIED ) THEN
loc_doc_class := loc_doc_class - web_global_var_pkg.CLASS_SECRET;
END IF;

-- Classification are 1-4 for non-nato and 5-8 for nato
-- This adjusts the class down to just 1-4 so I don't have to
-- keep making double checks
IF ( citation_class < title_class ) OR
( citation_class < identifiers_class ) OR
( citation_class < abstract_class ) THEN
   citation_class_error := 'Wrong Document Citation
Classification.';
out_status := -1;
END IF;

IF ( title_class > loc_doc_class ) THEN
   title_class_error :=
   'Title classification can NOT be higher than Document
classification.';
   document_class_error :=
   'Document classification can NOT be lower than Identifiers, ' ||
   'Abstract or Title.';