Statistical Analysis of Copper Doped Titanium Dioxide

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ABSTRACT

In this research, titanium dioxide and Al nanoparticles were purchased from a Chinese company, then the copper materials were added in three ratios that are (0, 1, 2, 3, and 5) wt.%. The use of dry press method through a hydraulic press from one direction, using a mold of diameter of 10 mm to obtain a ceramic compressed dimension that was firing at a temperature of 900 °C. The thermal conductivity is studied for all samples that include pure with the copper added proportions and compared between them when adding the proportions for copper the ratio of 2 wt.% is the highest conductivity among the used ratios. For the mechanical properties, which included the hardness when adding percentage from copper to TiO2 2 wt.% ratio showed is the highest hardness value between other ratios, the samples surface, taking surface pictures of the samples through an optical microscope.

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1. Introduction

TiO2 is the most abundant element, and it is one of the fifty-ninth minerals that make up the earth’s crust (0.6% of its mass). It is present in most igneous rocks and sediments and derived from them (as well as in living organisms and natural bodies of water). Of the 801 types of igneous rocks analyzed by the United States Geological Survey, its share in soils is about (0.5-1.5) % titanium.

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Titanium dioxide (or titanium dioxide) or titania is a naturally occurring oxide of titanium, with the chemical formula TiO$_2$. When used as a pigment, it is called Titanium White, Pigment White 6 Cl 77891. Titanium dioxide is popular for its many uses, from paint and sunscreen to food coloring. When used as a food coloring, it has the number (E171).

Application of titanium dioxide is a photocatalytic behavior [1-3], high-temperature energy devices [4], antibacterial activity [5], supercapacitor [6], solar cells [7], gas sensor [8, 9], for other applications for [10-49].

The goal of this project is to demonstrate the use of the dopant copper with the TiO$_2$ nanomaterials, then study the characterization of the materials obtained. The basic contributions in this thesis are enumerated as follows:

1. Preparation of Cu:TiO$_2$ (0, 1, 2, 3 and 5) wt.%.  
2. Investigate the mechanical properties (Hardness) of Cu:TiO$_2$ (0, 1, 2, 3 and 5) wt.%.  
3. Demonstrate the thermal properties of Cu:TiO$_2$ (0, 1, 2, 3 and 5) wt.%.  
4. Study the surface morphology of Cu:TiO$_2$ (0, 1, 2, 3 and 5) wt.%.

2. Experimental work  

2.1. Materials  

In this section, the practical part of the project will be presented, where it deals with a description of the materials and devices used in the analysis of the measurement of samples such as thermal conductivity, hardness, and optical microscope images.

The instruments have been used in the Table 1.

<table>
<thead>
<tr>
<th>No</th>
<th>Instrument</th>
<th>Model</th>
<th>Company</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Thermal Conductivity</td>
<td>Giffia, George Ltd</td>
<td>UK</td>
</tr>
<tr>
<td>2</td>
<td>Shore-D</td>
<td>ISO 14577-1</td>
<td>UK</td>
</tr>
<tr>
<td>4</td>
<td>HOT-Plate+ Magnetic Stärre</td>
<td>T2-HS390</td>
<td>UK</td>
</tr>
<tr>
<td>5</td>
<td>Analytical Balance</td>
<td>OHAUS</td>
<td>France</td>
</tr>
<tr>
<td>6</td>
<td>Hydraulic Press</td>
<td>XJU-145</td>
<td>UK</td>
</tr>
</tbody>
</table>

Samples were placed in the device between two temperature probes, with a temperature change measured through the thermometer placed in the device for an hour.

The amount of heat transferred through the specimen was calculated from the following equation

$$H = l \times V = \pi r^2 e (T_A + T_B) + 2 \pi r e [d_A (1/2) (T_A + T_B) + d_B T_B + d_C T_C]$$

(1)

where: $l \times V$: Thermal energy, which passed through the heating coil after reaching to equilibrium thermal state, $l$: Current (0.25 A), $V$: Voltage (6 V), Time (1 h), $e$: Temperature amount (thermal energy) passed through a unit area of disc material for each second ($W/m^2.K$), $d_A$, $d_B$, and $d_C$: thickness of the sample (mm), $T_A$, $T_B$, and $T_C$: temperatures of the disc (A, B and C) respectively, $r$: disc's radius (mm), $T_A$, $T_B$, $T_C$: temperatures of the disc (A, B and C), respectively.

From calculated ($e$), ($K$) can be calculated as in following equation:
\[ K = \left( \frac{T_A - T_B}{d_s} \right) = e^{\frac{T_A + 2}{r} \left( \frac{d_A + 1}{d_s} \right)} \left( \frac{T_A + 1}{(2d_s)} \right) d_s T_A \]

(2)

\( K \): coefficient of thermal conductivity for specimen (W/m.k).

The Shore Hardness meter was used to measure material hardness. Material hardness is defined as measuring material resistance to a permanent change in the shape when applying a force with hardness at constant pressure. Hardness is related to the soft deformation of the surface. There are mechanical properties related to hardness, such as soft strength and resistance to fatigue. Hardness testing scales are defined by ISO 14577-1.

The simplified optical wave path of a conventional optical microscope. The modern optical microscope is able to magnify an object by 1600 times with the 0.2 m limit in spatial resolution.

3. Mechanism of the work

1. The materials were mixed with ethanol of 99% purity and then placed on the magnetic mixing device to ensure homogeneity between the materials and prevent agglomeration occurring in the material.

2. After the mixing process, the mixture is placed in a drying oven at a temperature of 60 °C, after which the grinding process is done to obtain a homogeneous powder.

3. In this step, the dry press is used using a one-way hydraulic press with a pressure of 3 tons, after forming, coal was applied to the samples to reduce oxidation and the samples were placed in the oven at a temperature of 900 °C for two hours. As for the sintering process, the temperature was raised within two hours and kept at 900 °C.

4. Results and Discussion

Optical Microscope magnification power compound depends on the ocular and the objective lenses, the final magnification is 1600X. Figure 1 shows the surface by optical microscope to TiO\textsubscript{2} with percentage Cu (1, 2, 3 and 5) wt.% (1600X) respectively.

![Figure 1: Surface by optical microscope to TiO\textsubscript{2} with percentage Cu (0, 1, 2 and 3) wt.% (1600X) respectively.](image)

Through a light microscope, the surface of a material TiO\textsubscript{2} pure was observed. The homogeneity of the particles with each other and that the surface did not contain cracks, when adding the proportions of Al to TiO\textsubscript{2}, note that there is Cu particles that are molten and linked with TiO\textsubscript{2} particles, which means that there is a strong bond between TiO\textsubscript{2} and Al practical, which reduces the cracks and holes in the material.

In the hardness test, the material’s ability to be implemented or scratched is evaluated, and its hardness is measured. Generally, the hardness is greatly improved by decreasing \( d \), the shore diameter is a device for measuring the hardness, higher numbers on the scale indicate a greater resistance to indentation and thus harder materials.
Lower numbers indicate less resistance and softer materials. Table 2 shows the hardness results obtained within Shore during the extra hard range and the highest hardness is TiO$_2$. Figure 2 shows the relationship between TiO$_2$, the percentage of Cu added to it and hardness.

**Table 2: The values of the change in hardness after adding Cu (0, 1, 2, 3) wt.%.**

<table>
<thead>
<tr>
<th>Samples (%)</th>
<th>Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiO$_2$ pure-0</td>
<td>90.2</td>
</tr>
<tr>
<td>Cu:TiO$_2$-1</td>
<td>87.9</td>
</tr>
<tr>
<td>Cu:TiO$_2$-2</td>
<td>86.5</td>
</tr>
<tr>
<td>Cu:TiO$_2$-3</td>
<td>82.4</td>
</tr>
</tbody>
</table>

![Figure 2: Hardness values of Cu:TiO$_2$ samples.](image)

Table 3 shows the results obtained through equation 1 and 2 the temperature passing through the material was measured to measure the material's ability of thermal conductivity and that highest heat conductivity is the 3% (wt) ratio. Figure 3 shows the relationship between TiO$_2$, the percentage of Cu added to it and thermal conductivity.

**Table 3: The values of the change in thermal conductivity after adding Cu (0, 1, 2, 3) wt.%.**

<table>
<thead>
<tr>
<th>Sample (%)</th>
<th>$T_a$°C</th>
<th>$T_b$°C</th>
<th>$T_c$°C</th>
<th>$d_s$(mm)</th>
<th>$e$ (W/m$^2$.°C)</th>
<th>$K$(W/m.°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu:TiO$_2$-0</td>
<td>28</td>
<td>30</td>
<td>28</td>
<td>1.30</td>
<td>6.551291436</td>
<td>0.203663538</td>
</tr>
<tr>
<td>Cu:TiO$_2$-1</td>
<td>32</td>
<td>33</td>
<td>33</td>
<td>1.90</td>
<td>7.067890337</td>
<td>0.203663538</td>
</tr>
<tr>
<td>Cu:TiO$_2$-2</td>
<td>29</td>
<td>32</td>
<td>32</td>
<td>1.88</td>
<td>6.249048887</td>
<td>0.406212989</td>
</tr>
<tr>
<td>Cu:TiO$_2$-3</td>
<td>28</td>
<td>30</td>
<td>28</td>
<td>1.40</td>
<td>6.617801232</td>
<td>0.102893198</td>
</tr>
</tbody>
</table>
Figure 3: Thermal conductivity of Cu:TiO$_2$ after adding Cu (0, 1, 2, 3) wt.% samples.

In Figure 3, the thermal conductivity property of the material and Copper additives is shown: it was noticed through the results that Titanium dioxide nanoparticles were less conductive of heat between the ratios used because the thermal conductivity decreases with the decrease of the material particle size. The mechanism of heat transfer by electrons was more efficient than the contribution of a phonon. The 2 wt.% ratio was considered the highest thermal conductivity.

4.1. Standard Deviation (SD)

In science, it is typical to publish both the standard deviation of the data and the standard error of the estimate. Effects that deviate by more than two standard deviations from the null hypothesis are regarded as statistically significant and serve as a safeguard against erroneous conclusions that are really the result of measurement mistakes in random samples. To measure the extent of statistical dispersion and the most widely used among the measures of statistical dispersion, the standard deviation of a variable was calculated from the arithmetic mean using the following relationship:

$$\bar{x} = \frac{\sum x}{N} = \frac{x_1 + x_2 + \ldots + x_N}{n} = \frac{347}{4} = 86.75$$

(1)

$$\bar{x} = \frac{\sum x}{N} = \frac{x_1 + x_2 + \ldots + x_N}{n} = \frac{26.48603189}{4} = 6.621507973$$

(2)

$$\bar{x} = \frac{\sum x}{N} = \frac{x_1 + x_2 + \ldots + x_N}{n} = \frac{0.916433263}{4} = 0.229108316$$

(3)
where $\bar{x}$ the mean or mean, $x$ the percentage of addition, $N$ the number of samples prepared.

Table 4, 5 and 6 represent the SD of the values for Tables 2 and 3.

**Table 4: The SD of the sample's value for Hardness measurement.**

<table>
<thead>
<tr>
<th>x</th>
<th>$x - \bar{x}$</th>
<th>$(x - \bar{x})^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>90.2</td>
<td>3.45</td>
<td>11.9025</td>
</tr>
<tr>
<td>87.9</td>
<td>1.15</td>
<td>1.3225</td>
</tr>
<tr>
<td>86.5</td>
<td>-0.25</td>
<td>0.0625</td>
</tr>
<tr>
<td>82.4</td>
<td>-4.35</td>
<td>18.9225</td>
</tr>
</tbody>
</table>

In the latter, you write the deviation relationship as follows:

$$\sigma = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x - \bar{x})^2} = 2.837692725$$

**Table 5: The SD of the sample's value for e measurement.**

<table>
<thead>
<tr>
<th>x</th>
<th>$x - \bar{x}$</th>
<th>$(x - \bar{x})^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.551291436</td>
<td>-0.070216537</td>
<td>0.004930362</td>
</tr>
<tr>
<td>7.067890337</td>
<td>0.446382364</td>
<td>0.199257215</td>
</tr>
<tr>
<td>6.249048887</td>
<td>-0.372459086</td>
<td>0.138725771</td>
</tr>
<tr>
<td>6.617801232</td>
<td>-0.003706741</td>
<td>1.37399E-05</td>
</tr>
</tbody>
</table>

In the latter, you write the deviation relationship as follows:

$$\sigma = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x - \bar{x})^2} = 0.292799884$$

**Table 6: The SD of the sample's value for k measurement.**

<table>
<thead>
<tr>
<th>x</th>
<th>$x - \bar{x}$</th>
<th>$(x - \bar{x})^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.203663538</td>
<td>-0.025444778</td>
<td>0.000647437</td>
</tr>
<tr>
<td>0.203663538</td>
<td>-0.025444778</td>
<td>0.000647437</td>
</tr>
<tr>
<td>0.406212989</td>
<td>0.177104673</td>
<td>0.031366065</td>
</tr>
<tr>
<td>0.102893198</td>
<td>-0.126215118</td>
<td>0.015930256</td>
</tr>
</tbody>
</table>

In the latter, you write the deviation relationship as follows:

$$\sigma = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (x - \bar{x})^2} = 0.110217053$$

5. Conclusion

1. After explaining the process mechanism and working on the results, they can be summarized in the conclusion
2. Titanium dioxide and Cu materials were purchased from a Chinese company.
3. The proportions of Cu (0, 1, 2, 3, 5) wt.% respectively, were added to titanium dioxide.
4. The hardness of the samples was measured and it was noticed that the TiO$_2$ pure sample has the highest hardness and that the highest hardness of the added proportions of the Cu material is 2 wt.%.
The surface of the sample was observed by optical microscopy. It was found that the metal particles were dissolved between the TiO$_2$ particles, it was observed with an optical microscope at a capacity of 1600X magnification.

References


[49] Gharbi, S., R. Dhahri, M. Rasheed, E. Dhahri, R. Barille, M. Rguti, A. Tozri, and Mohamed R. Berber. "Effect of Bi substitution on nanostructural, morphologic, and electrical behavior of nanocrystalline La1-xBixNi0.5Ti0.5O3 (x=0 and x=0.2) for the electrical devices" Materials Science and Engineering: B 270 (2021): 115191.

[50]