CHAPTER 4

CHARACTERIZATION OF MECHANICAL PROPERTIES

4.1 INTRODUCTION

The study refers to the determination of the material properties through tests conducted on specimens designed for the purpose, as per standard procedures. The evaluated properties can be used for design and fabrication of industrial components for various applications. The mechanical properties of the coated specimens depend on the coated materials, the substrate and the coating method adopted.

The present study is concerned with the investigation on the effect of tempering of the substrate material and also the effect of using a bond coat before applying the top coat. Various mechanical properties as listed below were evaluated as per ASTM/ISO standards for the coated specimens. The behavior of the materials under various loading conditions was analyzed.

1. Tensile strength of the coating and the substrate system
2. Tensile strength of coating
3. Micro hardness
4. Adhesion
5. Coating thickness
4.2 EVALUATION OF TENSILE STRENGTH

The tensile specimens were tested in Instron tensile testing machine of 200kN capacity. The tensile strength, UTS (Ultimate tensile strength) and ductility have been measured by conducting static tension test in accordance with ISO-527. The bare and coated specimens and the geometry of the tensile test bars are shown in Figures 4.1 to 4.3.

![Figure 4.1 Tensile test bars](image1)

![Figure 4.2 Tensile test bars duplex coated](image2)

![Figure 4.3 Geometry of tensile test bar](image3)
Specimens were held in the hydraulic grips at a pressure of 600 psi (4.2 MPa). Load cell of 50 kN was selected for this test. The specimens were loaded in tension at a constant stroke rate of 2 mm/min.

4.2.1 Results and Discussion

Table 4.1 shows the results of the tensile tests conducted on standard specimens. Coating tensile tests are normally conducted using tubular fixture, to test the tensile strength of the coating. An attempt was made to test the coated samples as per ISO-527 to check if the coating affects the tensile properties. Literature reports, though few in this aspect, have confirmed the improvement in the tensile strength of coated specimens.

1. Yield stress values are higher for the T6 treated specimens duplex coated, with uncoated soft specimens having the lowest value.

2. Rupture stress values for the T6 treated specimens are highest with the specimens with duplex coating having higher values than the specimens with single layer mullite coating alone. The uncoated T6 treated specimens have an equivalent strength.

3. T6 treated specimens have higher corresponding values than soft samples.

4. Ductility values are lower for the uncoated specimens and higher for the other coated specimens.

5. Generally duplex coated specimens show better results.
Table 4.1 Tensile test results of coated and bare specimens

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Description of specimen</th>
<th>Yield stress (MPa)</th>
<th>Rupture stress (MPa)</th>
<th>Ductility (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Uncoated soft</td>
<td>132</td>
<td>204</td>
<td>4.5</td>
</tr>
<tr>
<td>2</td>
<td>Uncoated soft</td>
<td>130</td>
<td>218</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>Uncoated hard</td>
<td>169</td>
<td>288</td>
<td>4</td>
</tr>
<tr>
<td>4</td>
<td>Uncoated hard</td>
<td>172</td>
<td>293</td>
<td>4.5</td>
</tr>
<tr>
<td>5</td>
<td>Soft with single layer mullite coating</td>
<td>175</td>
<td>171</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>Soft with single layer mullite coating</td>
<td>165</td>
<td>170</td>
<td>4.8</td>
</tr>
<tr>
<td>7</td>
<td>Soft with duplex coating</td>
<td>175</td>
<td>175</td>
<td>5</td>
</tr>
<tr>
<td>8</td>
<td>Soft with duplex coating</td>
<td>173</td>
<td>174</td>
<td>4</td>
</tr>
<tr>
<td>9</td>
<td>Hard with single layer mullite coating</td>
<td>175</td>
<td>178</td>
<td>5.4</td>
</tr>
<tr>
<td>10</td>
<td>Hard with single layer mullite coating</td>
<td>173</td>
<td>177</td>
<td>4.5</td>
</tr>
<tr>
<td>11</td>
<td>Hard with duplex coating</td>
<td>259</td>
<td>274</td>
<td>4.7</td>
</tr>
<tr>
<td>12</td>
<td>Hard with duplex coating</td>
<td>214</td>
<td>275</td>
<td>6</td>
</tr>
</tbody>
</table>

Note: Hard refers to T6 treatment, single layer coating refers to coating without bond coat, duplex coating refers to coating with bond coat.

The yield stress and rupture stress values are shown in the Figures 4.4 and 4.5 for comparisons to be made.

![Yield stress values in MPa](image_url)

Figure 4.4 Yield stress values in MPa
4.3 EVALUATION OF TENSILE STRENGTH OF DUPLEX COATING

A simple and fairly reliable test for coating tensile strength is based upon two tubes, of the same diameter and accurately machined to give mating faces. Figures 4.6 and 4.7 show the coated samples and the Schmidt test setup, respectively. The tubes are mounted on a mandrel and sprayed along their length. The ends of the tubes are threaded to fit a tensile testing machine and after removal of the mandrel, the coating may be pulled to failure. Table 4.2 shows the results of the test and Figure 4.8 shows the bar graph for comparisons.
4.3.1 Results and Discussion

Generally the tensile strength of ceramic coatings are low as reported in the literature. Ceramics are weak in tension but strong in compression. For example, alumina has a tensile strength of 1100MPa, while the compressive strength is 2400 MPa. The reason is due to the brittle nature of ceramics and also due to the internal flaws present in ceramics, from which cracks propagate in tension, but not in compression. Coating tensile strengths of functionally graded coatings are reported to be around 100 MPa, due to improved microstructure of the coatings.

Table 4.2 Coating tensile strength measurements

<table>
<thead>
<tr>
<th>Trial No.</th>
<th>Coating tensile strength in MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>50</td>
</tr>
<tr>
<td>2</td>
<td>52</td>
</tr>
<tr>
<td>3</td>
<td>47</td>
</tr>
<tr>
<td>4</td>
<td>45</td>
</tr>
<tr>
<td>5</td>
<td>48</td>
</tr>
</tbody>
</table>
4.4 EVALUATION OF ADHESION STRENGTH OF COATINGS

4.4.1 Introduction

A major requirement of any coating is that the adhesion to the substrate is sufficient to maintain its integrity during use and the measurement of coating adhesion is therefore of major practical importance. Several reviews have already been written on the subject of adhesion measurement (Davies and Whittaker 1967, Jacobeson 1976, Mittal 1976, Kretzschmar 1977, Christopher Berndt 1980). Comparison of the adhesive strength of duplex mullite coated specimens have been made with that of duplex coated and functionally graded YSZ coatings and with aluminum oxide coatings, and results presented.

4.4.2 Mechanisms of Adhesion

Possible mechanisms for the adhesion of plasma and flame sprayed coatings have been reviewed by Matting and Steffens (1967) and Christopher Berndt (1980) and are summarized below.
4.4.2.1 Mechanical keying

Mechanical keying results from the deposit material flowing round roughness peaks and into undercut regions of the substrate surface. This mode of adhesion explains the experimental observation that materials deposited onto polished substrates do not exhibit a high degree of adhesion; whereas the adhesion of the coating is markedly improved if the substrate is roughened.

4.4.2.2 Physical adhesion by dispersive forces

When two materials come into close contact some mutual attraction may be expected resulting from Vander wall’s forces. This mechanism presupposes that the materials in contact are only one lattice spacing distant from each other and also that the deposit material wets the substrate. Dispersion or Vanderwall’s forces predict the highest adhesive strengths. Dispersion forces do not depend on temperature and adhesion by Vanderwall’s forces takes place athermally.

4.4.2.3 Chemisorption and epitaxy

Chemisorption and epitaxy, applied to plasma spraying, concerns the oriented growth of the liquid onto the substrate. A surface roughening operation greatly reduces the energy barrier which must be overcome for oriented growth to occur because of the zones of high elastic lattice energy which are formed. At the same time macroscopic regions of oriented growth are unlikely due to the geometry of the substrate surface. The epitaxial growth of crystals is therefore expected to occur on a microscopic scale and this is confirmed from electron microscope studies.

4.4.2.4 Diffusion

If a metal is sprayed onto a metal substrate of the same material it is likely that self-diffusion will result. Also heated regions of plastically
deformed areas of the substrate can recrystallise after contact with the sprayed particles and such zones are generally only a few microns deep. Since the substrate has a high defect concentration due to cold working and the splat cooled deposit material a high vacancy concentration, equivalent to that at high temperatures, the diffusion coefficients of both materials would increase greatly and enhanced interdiffusion could occur.

4.4.2.5 Reaction

An intermediate phase may be produced simultaneously with diffusion. For example, Fe₂MO₆ is formed at the high energy roughness peaks of a mild steel substrate when sprayed with molybdenum. Another example is the formation of intermetallic phases (Fe₃Al, FeAl) at the interface when aluminum is sprayed onto steel.

In the present thesis all the above mechanisms are likely to occur in the coating and contribute for the coating adhesive strength.

4.4.3 Factors Affecting Adhesion Strength

4.4.3.1 Deposit thickness

Some results of coating strength in relation to coating thickness are discussed below (Lyashenko et al 1969 and Sharivker 1967). There were three regions of interest. In the thickness range greater than 0.7 mm adhesive failure took place and the experimental points fitted a straight line with a negative slope. Within a coating thickness range of 0.45 to 0.70 mm a mixed adhesive –cohesive failure occurred. While for thicknesses between 0.3 to 0.5 mm the failure was reported as pure cohesive and these represent the strength of the coating layer. Zakharov et al (1970) on the other hand, found that the bond strength increased continuously with a decrease in coating thickness. He
established an empirical relationship of the relative bond strength and coating thickness for the case of stabilized zirconia deposited onto a steel substrate.

\[ \frac{\sigma}{\sigma_a} = 0.8/e^{3.2hc} + 0.2 \]  \hfill (4.1)

where, \( \frac{\sigma}{\sigma_a} \) = the relative adhesive strength
\( \sigma \) = measured adhesive strength of coating (MPa)
\( \sigma_a \) = maximum adhesive strength of coating (MPa)
\( h_c \) = coating thickness (mm)

Similar results can be expected in the case of mullite coated on aluminum. Hence a tradeoff is required between adhesion strength and thermal shock resistance, as higher thickness of the coating will improve the thermal barrier properties of the coating. The recommended coating thickness of 0.3 to 0.5 mm was used in the thesis which will give dual benefits.

### 4.4.3.2 Profile of grit blasted substrate

There is general agreement that adhesion markedly depends upon the substrate surface preparation. For example, Apps (1974a, 1974b) found that the type and condition of the grit blasting medium and the angle of blasting influenced the bond strength of plasma sprayed aluminum coatings on steel. A rough surface with more irregularities are sights of mechanical keying and the adhesion strength will improve.

### 4.4.3.3 Residual stresses

Another important factor influencing coating adhesion is the residual stress system developed as the coating solidifies and cools. Residual stresses have been estimated theoretically and also directly measured. These
studies have been concerned mainly with the influence of the physical properties of the coating and substrate materials (Thermal conductivity, thermal expansion coefficient and specific heat) on interfacial and surface residual stresses, and their effect on coating adhesion.

4.4.4 Inference on Adhesive Strength of Plasma Sprayed Coatings

It is apparent from the literature that there has been very little work carried out on the central problem of adhesion of plasma sprayed coatings, namely direct determination of the mechanism by which the coating is detached from the substrate and the manner in which this is influenced by the nature of the substrate surface, residual stresses in the coating, coating microstructure and interaction between the sprayed particles and substrate.

4.4.5 Adhesion Strength Measurement

The strength is found from the simple relation

\[ \sigma = \frac{N}{a}, \]

(4.2)

where, \( \sigma \) = cohesive or adhesive strength (MPa)

\( N \) = force at failure (N)

\( a \) = cross sectional area of base (m²)

ASTM C 633-01 standard test method for testing adhesion and cohesion strength of thermal spray coatings is explained below. Refer Figure 4.9 for the test set up.
Figure 4.9 ASTM C-633 adhesion testing principle

Cylindrical specimens were used to measure the adhesive strength. The coated cylindrical specimen, block A, was bonded to uncoated block B with epoxy glue to construct the tensile specimen. The adhesive strength was carried out on Instron tensile testing machine as per ASTM C 633-01 standards. 16 specimens were tested for the evaluation of adhesive bond strength, comprising of single layer mullite coated soft and T 6 treated specimens, and duplex coated soft and T 6 treated specimens. The test procedure is explained. A 25 mm diameter steel cylinder end is sprayed with the required coating. Another cylinder with the same diameter is glued on top of this, using a high temperature epoxy adhesive. The adhesive is cured in a muffle furnace at 180°C before application. The assembly is clamped and held for duration of 30 minutes till the adhesive sets. The clamp is removed, and then the pieces are mounted in a tensile testing machine using a special adhesion testing fixture. The force is applied by pulling them apart and the pull strength recorded. Usually this test method is performed at ambient temperature.

This test method is limited to testing thermal spray coatings that can be applied in thickness greater than 0.30 mm. The limitation is imposed
because an adhesive bonding agent is used in the test. Those bonding agents established so far for this method tend to penetrate thermal spray coatings and may invalidate results unless the coatings are thick enough to prevent penetration through the coating. Recommended strain rates during tensile testing are 0.013 to 0.021 mm sec\(^{-1}\).

The tensile adhesion test is simple to carry out and is useful in quality control and for providing a ranking of various types of coatings (Mock 1966, Gerdeman and Hecht 1972). The following points need consideration:

1. The adhesive that is used to attach the plug to the coating must have a greater tensile strength than the coating. These adhesives are not readily available and have a limited shelf life.

2. Flaws in the form of micro-cracks, porosity and second phase inclusions within the coating will affect the adhesion of the coating. The role of these micro structural features of the coating cannot be examined by the tensile adhesion test.

Figures 4.10 to 4.12 show the photograph of the test specimen, geometry of the coated specimen and the photograph of the test in progress respectively.

![Figure 4.10 Coated adhesion test specimens](image)
The results are shown for the T6 treated duplex coated specimens only. The adhesive strength was lower (10 to 14 MPa) in the case of other specimens due to softer substrates and poorly adhering single layer mullite coatings.

The adhesive strength results by tensile tests are listed in Table 4.3. In the tensile test process, the failure is almost the peeling of coatings from
the substrate, which shows that the combining interface between coating and substrate is the weakest point in the coating-substrate system. The bond coat has thermal expansion coefficient between that of mullite and aluminum and hence reduces the residual stresses in the coating system and improves the adhesive strength. Literature study reported a strength of 18.8 MPa for Al₂O₃ oxide coatings. The bar graph of the results is presented in Figure 4.13. A comparative study of adhesion measurements of duplex coated YSZ, functionally graded YSZ, functionally graded Ti-6Al-4V and Fe₃Al /Al₂O₃ with duplex coated mullite is presented in Table 4.4.

**Table 4.3 Adhesive bond strength results**

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Adhesion strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21</td>
</tr>
<tr>
<td>2</td>
<td>19.5</td>
</tr>
<tr>
<td>3</td>
<td>20.2</td>
</tr>
<tr>
<td>4</td>
<td>22</td>
</tr>
<tr>
<td>5</td>
<td>19.4</td>
</tr>
<tr>
<td>6</td>
<td>21.3</td>
</tr>
</tbody>
</table>

**Figure 4.13 Bar graph of adhesion strength measurements**
Table 4.4 Comparison of mullite coatings with FGC’s

<table>
<thead>
<tr>
<th>Description</th>
<th>Yttria stabilized zirconia/NiCoCrAlY</th>
<th>Hydroxyapatite/Ti-6Al-4V FGC</th>
<th>Fe$_3$Al/Al$_2$O$_3$ FGC</th>
<th>Mullite coated cast Al (Duplex Coating)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bond Strength in MPa</td>
<td>9.3</td>
<td>17.8</td>
<td>38</td>
<td>51</td>
</tr>
</tbody>
</table>

4.5 EVALUATION OF MICROHARDNESS

Small specimens are sliced from the coated samples. Samples containing coating cross sections are mounted and polished for the micro hardness measurement. Microscopic observation under optical microscope of the polished section of the coatings exhibits three distinctly different regions/phases namely grey, dark and spotted/mixed. Vickers Micro hardness measurement is made on these optically distinguishable phases using HX-1000 Micro hardness tester equipped with a monitor and a microprocessor based controller, with a load of 100gf and a loading time of 20 seconds. About twelve or more readings are taken on each sample and the average value is reported as the data point. The hardness is due to the presence of oxides and this is confirmed by XRD analysis.

4.5.1 Results and Discussion

Figure 4.14(A, B, C) shows the distribution curve of micro-hardness along the thickness of coatings and the micrographs and SEM images of the various phases. The variation in the hardness in the coating can be attributed to distinct phases of NiCr, mullite and the interface between the bond and top coats. NiCr bond coat has a hardness of 600 HV (Powder Alloy Corporation, USA) and the mullite top coat has a hardness of 1000 HV (Wolfgang & Schneider, 1989). The hardness at the substrate/bond coat interface was
measured as 500 HV and at the surface was 1000 HV. There was a gradual increase in the hardness of the coated layers from 500 HV to 1000 HV, due to the two phases of NiCr and mullite.

Figure 4.14 (A) Micro-hardness profile

Structurally different phases bear different ranges of hardness that may depend on different phase’s present/formation in the coating, which is clear from X-ray diffraction analysis. Micro-hardness measurement is made on optically distinguishable phases present in the coatings. The existences of at least three different phases (which are optically distinguishable) have been formed during plasma spraying. The hardness values are different for different phases. On referring to the XRD patterns, it becomes evident that, during coating deposition, oxidation and transformation of phases have taken place. Average values are shown in the micro hardness profile.
4.6 EVALUATION OF COATING THICKNESS

The thickness of the coating plays a major role in thermal barrier coatings. As already highlighted, higher the thickness, better will be the thermal barrier properties, but a compromise has to be made on the mechanical strength of the coating, as a thinner coating has a better mechanical strength. In this study, a thick duplex coating of 300+ 50 µm has been used to enhance the thermal properties suitable for the application in this study. The mechanical properties have been evaluated and presented earlier
and found satisfactory for the intended application. The thickness of a thin layer on a surface can be measured by a large variety of methods as given in the Table 4.5.

**Table 4.5 Methods of coating thickness measurements**

<table>
<thead>
<tr>
<th>Method</th>
<th>Types</th>
</tr>
</thead>
<tbody>
<tr>
<td>Optical methods</td>
<td>Metallography, SEM, Holography</td>
</tr>
<tr>
<td>Removal methods</td>
<td>Coulometric, Feeler gauge, Ball cratering</td>
</tr>
<tr>
<td>Electromagnetic methods</td>
<td>Magnetic flux, Eddy current Capacity</td>
</tr>
<tr>
<td>Scattering methods</td>
<td>Beta back scattering, Ultrasonic</td>
</tr>
<tr>
<td>Excitation methods</td>
<td>X-ray fluorescence</td>
</tr>
</tbody>
</table>

For thickness measurements the ball crater method also is used. This method is simple, cheap and easy to use. The coating thickness was checked using a coating thickness gauge, machine number MI Coat 22, model TT 210 at an ambient temperature of 30°C. Six specimens with the geometry shown in Figure 4.15 were coated using optimized spray parameters, and the spray time was noted for each sample.

![Figure 4.15 Geometry of specimen for coating thickness measurement](image-url)
Coating thickness ranged from 350 to 400 μm for the duplex coating tested randomly on four locations on the surface of the samples on 6 specimens. The noted spray time and optimized spray conditions will be used for the coating of engine components, in this study. The results of the measurements are shown in Figure 4.16.

![Coating thickness results](image)

**Figure 4.16 Coating thickness results**

## 4.7 MODELING AND SIMULATION OF THE MECHANICAL BEHAVIOUR OF MULLITE COATED CAST ALUMINUM

### 4.7.1 Introduction

Simulation studies were conducted using FEM (Finite element method), to understand the mechanical behavior and the thermal behavior of three different coated models as detailed below.

1. Boron carbide plasma sprayed on cast aluminum A 356.0.
2. Mullite plasma sprayed on cast aluminum A 356.0 with bond coat of nickel chrome.
3. Mullite plasma sprayed on cast aluminum A 356.0 without bond coat of nickel chrome.
The above study helped in comparison of the performance of the coatings.

Modeling and Simulation of coating design before costly experimentation and bulk processing has always been an effective tool to arrive at a good design of the coating/substrate system. An attempt has been made in this study to simulate the mechanical behavior of boron carbide coatings and mullite coatings plasma sprayed on cast aluminum substrates, for comparison. A popular ceramic material for coating has been YSZ (8% Yttria stabilized zirconia) and few attempts have been made using boron carbide as a coating material by researchers. The displacement, stress plots for the standard bend test and tensile test using a nominal load of 50 N have been prepared for all models using ANSYS 8.0 software to arrive at a reasonably good conclusion to carry out validation experiments. Similarly temperature distribution plots have been prepared to study the heat transfer across the cross section of the coatings. Comparisons have been made and results arrived.

For this study boron carbide powder, a ceramic with high wear resistance and hardness, high modulus, poor thermal conductivity and light weight has been applied as an overlay coating on aluminum A 356.0, a cast alloy with light weight and compared to the ceramic material having a low modulus and high thermal conductivity. The purpose of this hard coating of up to 100 microns is to improve the wear properties and the thermal barrier property of aluminum, but maintaining the low weight of the coating/substrate system. Modeling and simulation of such a system has been studied. The tensile strength, the bend strength and the temperature distribution have been compared for the uncoated and coated models. FEA (Finite element analysis) is a good tool to study the stress and the strain of geometric models,
Similarly mullite duplex coatings with a bond coat of nickel chrome and single layer coating of mullite has been applied on cast aluminum A 356.0 for a coating thickness of 200 microns and 100 microns respectively, and the FEM simulation conducted.

The element used is Solid 95. SOLID 95 is a 3-D, 20-Node Structural Solid. SOLID 95 is a higher order version of the 3-D 8-node solid element (SOLID 45). It can tolerate irregular shapes without as much loss of accuracy. SOLID 95 elements have compatible displacement shapes and are well suited to model curved boundaries. The element is defined by 20 nodes having three degrees of freedom per node: translations in the nodal x, y, and z directions. The element may have any spatial orientation. The element has plasticity, creep, stress stiffening, large deflection, and large strain capabilities.

Figure 4.17 shows the coated model for the single layer coating and Figure 3.2 the duplex coating (shown in chapter 3). Figure 4.18 shows the tensile test and bend test loads applied on the coated specimens. The results are taken as deformation and stress plots.

![Coating Model](image)

**Figure 4.17 Coating model for single layer**
Figure 4.18 Tensile and bend test loads applied on the coated models

4.7.2 Methodology

The properties of the ceramic powders and the substrate used for the study are given in table 3.3. An aluminum plate of cast alloy A 353.0 and of size 50mm x 50 mm x 6 mm has been used. The top plate of area 50 mm x 50 mm is coated to a depth of 100 microns using 2 to 3 runs of plasma spraying with minimum overlap for the single layer coating and 200 microns for the duplex coating. The coating is assumed to be made and test specimens ready for testing. The coated models are simulated for the mechanical and thermal behavior. Table 4.6 shows the simulated result summary.

Table 4.6 Summary of the simulated results

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Test Description</th>
<th>Uncoated cast aluminum</th>
<th>BC coated cast aluminum</th>
<th>Duplex coating on cast aluminum</th>
<th>Single layer mullite coating on cast aluminum</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Bend test displacement in mm</td>
<td>0.064</td>
<td>0.033</td>
<td>4.455</td>
<td>4.445</td>
</tr>
<tr>
<td>2.</td>
<td>Bending stress in MPa</td>
<td>144.877</td>
<td>340.618</td>
<td>0.195 x 10^8</td>
<td>0.233 x 10^8</td>
</tr>
<tr>
<td>3.</td>
<td>Tensile test displacement in mm</td>
<td>0.0032</td>
<td>0.006</td>
<td>0.179 x 10^-3</td>
<td>8.84 x 10^-5</td>
</tr>
<tr>
<td>4.</td>
<td>Tensile stress in MPa</td>
<td>14.193</td>
<td>25.392</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>5.</td>
<td>Temperature distribution in °C from top coated layer to bottom layer of cast aluminum</td>
<td>600 to 105</td>
<td>600 to 67</td>
<td>600 to 67</td>
<td>600 to 87</td>
</tr>
</tbody>
</table>
4.7.3 Results and Discussion of the Simulation Study

1. Referring to the tensile test plots, it is clear that the tensile stresses are higher in the boron carbide coated samples and the mullite coated samples than in the uncoated samples owing to the fact that there is a wide difference in the modulus values of boron carbide/mullite and aluminum. But the boron carbide coating was better than the mullite coating due its higher elastic modulus. However the results are poor for the coated models, which confirm and corroborate the results highlighted in the literature survey. The wide difference in the mechanical and thermal properties leads to delamination cracking of the coating (Bao and Cai 1997).

2. The bend test plots show a poor mechanical behavior of both the coated models when compared to the uncoated model. But the boron carbide coated model shows a better result when compared to the mullite coated model, owing to its higher flexural strength.

3. The temperature plots show a more even distribution of the temperature for the uncoated model in comparison to the coated model but the heat loss is less in the coated model than the uncoated model, which is encouraging. The ceramic can be used as a thermal shield.

4. Mullite coatings as well as boron carbide coatings are poor in mechanical properties but the thermal barrier properties are good.

5. Cost is the limitation in the case of boron carbide coatings, when compared to mullite coatings.
6. In another study by the Viswanath A 2 (2009), boron carbide powder, a ceramic with high wear resistance and hardness, high modulus, poor thermal conductivity and light weight has been applied as an overlay coating on aluminum 6061, a wrought alloy with light weight and compared to the ceramic material, having a low modulus and high thermal conductivity. The purpose of this hard coating of up to 100 microns is to improve the wear properties and the thermal barrier property of aluminum, but maintaining the low weight of the coating/substrate system. The simulation study revealed poor mechanical properties of the coating, which can be improved by changing the coating design to duplex or multilayer. However the temperature plot revealed that the coating can be used as thermal shield as there will be less heat loss.

4.8 STATISTICAL ANALYSIS

4.8.1 Statistical Analysis of Tension Test

Statistical analysis has been performed using analysis of variance one-way ANOVA, which considers the effect of one controlled parameter (factor) upon the performance of the process. In this work the factor investigated is the effect of type of specimen (soft or tempered, with or without bond coat and two different levels of coating thickness applied) on the mechanical properties. The experimental data from tension tests is presented in the Table 4.7. Tests were conducted on cast aluminum A 356.0, both soft and T 6 treated.
Table 4.7 Tensile strength in MPa from experiments

<table>
<thead>
<tr>
<th>Type of material (Coating thickness is nominal)</th>
<th>Total of observations $y_i$</th>
<th>Averages $y_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soft</td>
<td>792</td>
<td>158.4</td>
</tr>
<tr>
<td>Soft with single layer coating of mullite (150 µm thick)</td>
<td>860</td>
<td>172</td>
</tr>
<tr>
<td>Soft with duplex coating (300 µm thick)</td>
<td>897</td>
<td>179.4</td>
</tr>
<tr>
<td>T6 treated</td>
<td>1443</td>
<td>288.6</td>
</tr>
<tr>
<td>T6 treated with single layer coating of mullite (150 µm thick)</td>
<td>1511</td>
<td>302.2</td>
</tr>
<tr>
<td>T6 treated with duplex coating (300 µm thick)</td>
<td>1557</td>
<td>311.4</td>
</tr>
</tbody>
</table>

Since six different material types and in each case five specimens were tested, there are six levels (or treatments) and five observations at each level. The objective is to test the appropriate hypothesis about the treatment and to estimate them. The results of the statistical analysis for the tensile properties of the specimens are presented in Table 4.8.

Table 4.8 Analysis of variance for tensile strength data

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Sum of squares</th>
<th>DOF</th>
<th>Mean square</th>
<th>Ratio of Sample Variance, $F_0$</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material type</td>
<td>130765.06</td>
<td>5</td>
<td>26153</td>
<td>1377.6</td>
<td>&lt; 0.05</td>
</tr>
<tr>
<td>Error</td>
<td>455.64</td>
<td>24</td>
<td>18.98</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>131220.7</td>
<td>29</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

It is seen from the above tables that between treatment mean squares are many times larger than within treatment or error mean square. The F ratios for the tensile strength are computed as $F_0 = 26153/18.98 = 1377.6$ and compared with an appropriate upper tail percentage point of $F_{5,24}$
distribution. For 5 % level of significance (risk), \( F_{0.05, 5, 24} = 2.62 \) from statistical table (Douglas C. Montgomery 1997). Since \( F_0 \) for tensile strength is greater than 2.62, the difference in material type (hardness of substrate, bare, single layer and duplex layer of coating), significantly affects the mean tensile strength and it can be concluded that an upper bound for the P-value is 0.05; that is \( P < 0.05 \). Since the factors involved are qualitative in nature, regression analysis is not made.

4.8.2 Statistical Analysis of Adhesion Test

The experimental data for the adhesion test is presented in the Table 4.9. Tests were conducted on cast aluminum A 356.0 specimens, soft and T 6 treated. Since eight different material types and in each case three specimens were tested, there are eight levels (or treatments) and three observations at each level. The objective is to test the appropriate hypothesis about the treatment and to estimate them. The results of the statistical analysis for the adhesion properties of the specimens are presented in Table 4.10.

**Table 4.9 Adhesion strength in MPa from experiments**

<table>
<thead>
<tr>
<th>Type of material (Coating thickness is nominal)</th>
<th>Total of observations ( y_i )</th>
<th>Averages ( \bar{y}_i )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soft with single layer mullite coating (100 µm thick)</td>
<td>32</td>
<td>10.7</td>
</tr>
<tr>
<td>Soft with single layer mullite coating (150 µm thick)</td>
<td>29.5</td>
<td>9.8</td>
</tr>
<tr>
<td>Soft with duplex coating (200 µm thick)</td>
<td>36.8</td>
<td>12.3</td>
</tr>
<tr>
<td>Soft with duplex coating (300 µm thick)</td>
<td>34.8</td>
<td>11.6</td>
</tr>
<tr>
<td>T 6 treated with single layer mullite coating (100 µm thick)</td>
<td>42.7</td>
<td>14.2</td>
</tr>
<tr>
<td>T 6 treated with single layer mullite coating (150 µm thick)</td>
<td>40.3</td>
<td>13.4</td>
</tr>
<tr>
<td>T 6 treated with duplex coating (200 µm thick)</td>
<td>58.9</td>
<td>19.6</td>
</tr>
<tr>
<td>T 6 treated with duplex coating (300 µm thick)</td>
<td>54.7</td>
<td>18.2</td>
</tr>
<tr>
<td>( \bar{y}_n = 329.7 )</td>
<td>( \bar{y}_n = 13.725 )</td>
<td></td>
</tr>
</tbody>
</table>

**Note:** The coating thickness varied from 100 to 150 µm for single layer coating and 200 to 300 µm for the duplex coating.
Table 4.10 Analysis of variance for adhesion strength data

<table>
<thead>
<tr>
<th>Source of variation</th>
<th>Sum of squares</th>
<th>DOF</th>
<th>Mean square</th>
<th>Ratio of Sample Variance, $F_0$</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material type</td>
<td>260.1</td>
<td>7</td>
<td>37.2</td>
<td>744</td>
<td>&lt; 0.05</td>
</tr>
<tr>
<td>Error</td>
<td>0.9</td>
<td>16</td>
<td>0.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>261</td>
<td>23</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

It is seen from the above tables that between treatment mean squares are many times larger than within treatment or error mean square. The F ratios for the adhesion strength is computed as $F_0 = 37.2/0.05 = 744$ and compared with an appropriate upper -tail percentage point of $F_{7,16}$ distribution.

For 5% level of significance (risk), from statistical table (Douglas Montgomery 1997), $F_{0.05, 7, 16} = 2.66$. Since $F_0$ for the adhesion strength is greater than 2.62, the difference in material type (hardness of substrate, single and duplex layer coating), significantly affects the mean adhesion strength and it can be concluded that an upper bound for the P-value is 0.05; that is $P < 0.05$.

4.9 CONCLUSION

The mechanical behavior is found satisfactory for the duplex coating system with bond coat and age hardened substrate (T6 treatment), especially the tensile strength of the coating/substrate system (275 MPa), adhesive strength (20 MPa), and micro hardness (1000 HV) for applications in which low loads act on components in service. But the coating tensile strength is low (50 MPa) as expected for ceramic materials, which can be accepted for applications where low tensile loads act on the components, for example IC engines. There is wide scope for improving the mechanical properties by controlling the process and designing the coating to avoid the wide mismatch in the properties. One such technique which can be used is to
build a functional gradient across the cross section of the coating. The FEA analysis has confirmed the expected results of the coating prior to experimentation and hence all the deficiencies encountered can be corrected and an efficient coating can be produced. Moreover, for an IC engine application, since mechanical loads are low on the piston crown when compared to the thermal load, thermal barrier property and residual stresses are more important. This is achieved by using mullite due its high creep resistance and thermal shock strength and also due to its low cost and availability. The thermal barrier properties of ceramic coating substrate system is generally good and can be used in many applications requiring low weight, wear resistance and hardness of the surface and exposure to high temperature. Statistical analysis was conducted for the tensile and adhesion strengths of different types of specimens and found that T 6 treatment and duplex coating improves the mechanical properties of the coated substrates and also the analysis of variance (ANOVA), confirmed the variation in the measured values.