CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials

Two types of metal alloys were used in this study:

1) Commercial pure titanium ingots (Easyti system®, Italy).
2) Cobalt-chromium ingots (Wirobond LFC BEGO, Germany).

3.1.1 Commercial pure titanium grade II (CPTi II)

Pure titanium ingots (Easyti system®, Italy) used in this study was conformed to the standards of European directives and Nordic institute of dental materials (NIOM) and certified by the Scandinavian Institute of Dental Materials (Figure 3.1).

Figure 3.1 Commercial pure titanium ingots (Easyti system®, Italy)

Table 3.1 Commercial pure titanium grade II alloy components (Wirobond LFC BEGO, Germany)

Elements	% In CPTi II
Ti	99.57
O	0.25
Fr	0.03
H	0.015
C	0.10
N	0.03

3.1.2 Cobalt-chromium alloy (Co-Cr)

Cobalt-chromium ingots (Wirobond LFC BEGO, Germany) were shown in Figure 3.2.

Figure 3.2 Cobalt-chromium ingots (Wirobond LFC BEGO, Germany)

Table 3.2 Cobalt-chromium compositions (Wirobond LFC BEGO, Germany)

Element	% In the alloy
Co	33
Cr	30
Ni	0.2
Fe	29
Mo	5
Mn	1.5
Si	1
C	0.3

3.2 Method

3.2.1 Titanium (CPTi)specimens preparation

3.2.1.1 Preparation of mould

Fifteen plastic models designed with conical shoulder shape and with the dimensions of 1mm thickness ×3 width ×42 length mm (according to the dental base metal alloys (ISO 6871-1)) were prepared as illustrated in Fig. 3.3 and Fig.3.4.

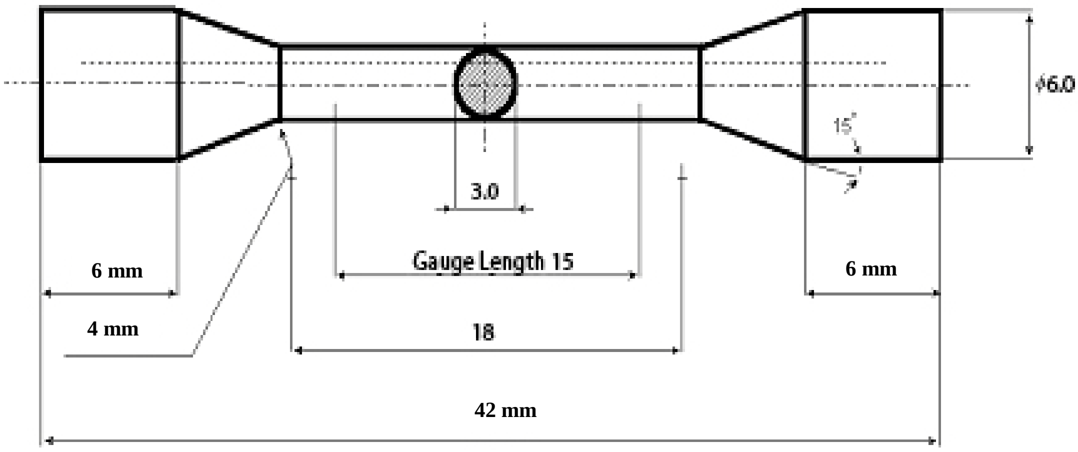

Figure 3.3 Schematic representations of the shape and dimensions of the study specimen (titanium/cobalt-chromium).

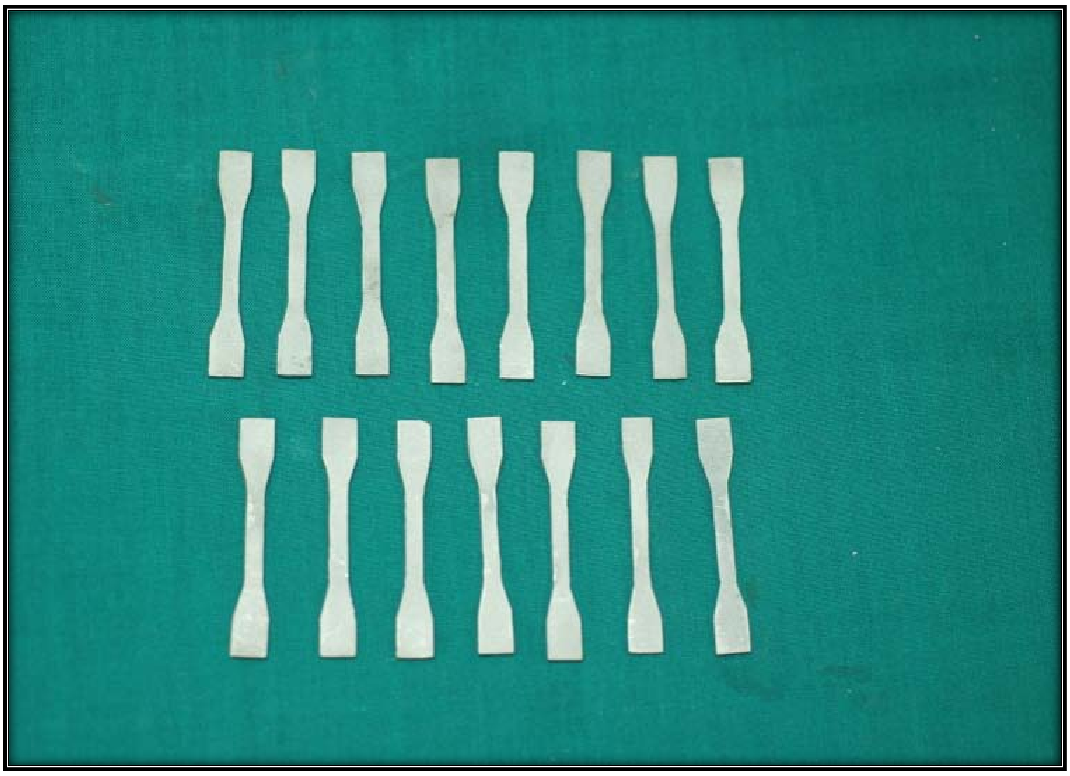

Figure 3.4 Shape of titanium specimens.

3.2.1.2 Titanium investing

The plastic model were invested and moulded into mould rings with a special silica bonded investment material for titanium (TICOAT S+L) available in 5 kg powder packs (10 bags of 500 g Each), with a fluid bottle of 770 cc capacity (Figure 3.5).

Figure 3.5 Investment materials used in the study for casting titanium specimen (TICOAT S+L)

100g TICOATS amount of powder with 15 cc TIEOAT L liquid were added. The investment materials initially were spatulated before mixed in the mixing machine as recommended by the manufacturer. The investment materials were then poured slowly in the ring while putting the mould ring on the vibrator in order to get rid of the air bubbles. This procedure was done 45 minutes at the room temperature until thoroughly hardened before casting.

3.2.1.3 Titanium casting technique

3.2.1.3.1 Heating cycle

The rings were placed in the oven at the room temperature. In a rising speed of 7°C per minute, primary heating temperature of the oven was set to 260 °C for 45 min until burn out. Afterward, heating temperature was slowly increased to 1100°C and kept for 45 minutes. The temperature then set back to 200°C starts casting after 60 min pause at 200°C temperature.

3.2.1.3.2 Casting

The casting machine used in the study (Manfredi Neutrodyn Easyti, Italy) (Figure 3.6).The casting technique used in this study was the arc melting technique under Argon protection, and casting by centrifuge under vacuum.

Figure 3.6 Casting machine.

The mould ring was placed on the casting machine arm, and the titanium ingots put in the centre of the machine inside a single-use special coated ceramic crucible (C15TI) as shown in Figures 3.7 and 3.8.

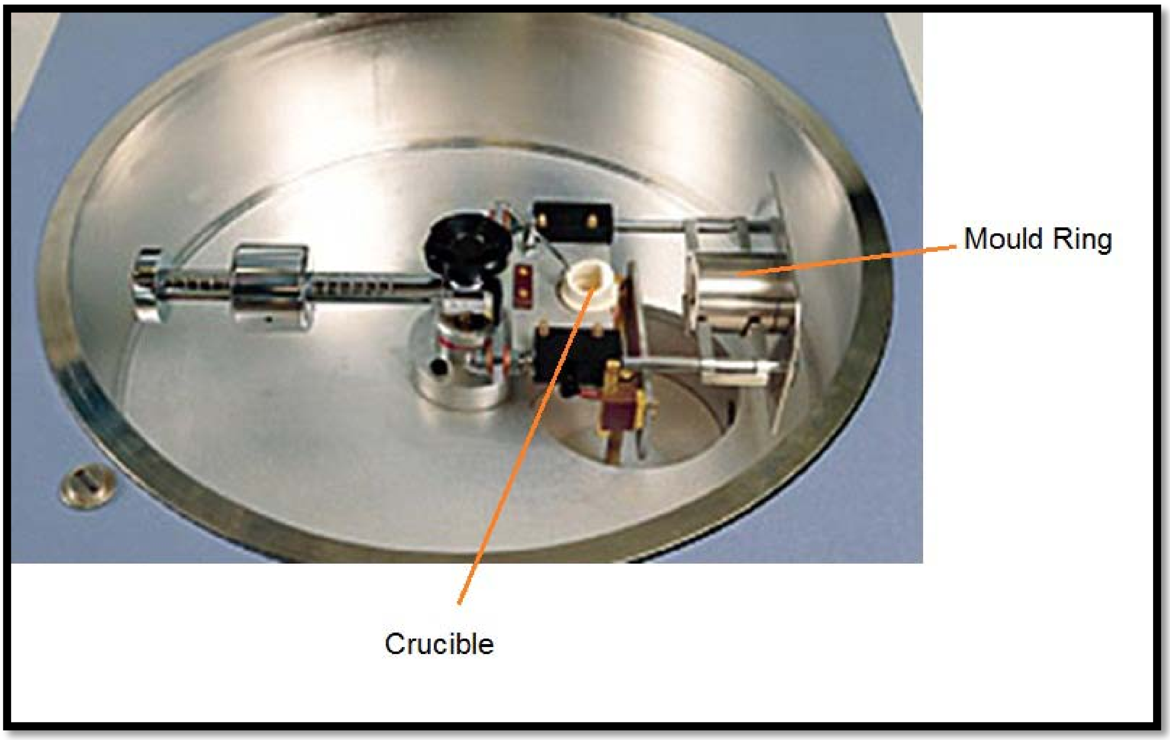

Figure 3.7 Crucible and mould ring placed in casting machine chamber.

Figure 3.8 Ceramic crucible (C15TI).

Before the centrifuge starts, the Argon gas was applied to the chamber and vacuumed for five times to clean the casting chamber atmosphere from any oxidation gas. The casting machine temperature was set to 1700^{o}C and started heating until the titanium ingots melt completely. After that, the centrifuge process started for 15 seconds to allow the shooting of the melted titanium inside the ring. After casting finished, rings were set aside at room temperature to cool down.

3.2.1.3.3 Polishing

Each cast specimen was then retrieved from the investment, sandblasted using 50µm silicon dioxide (SiO_2) straps at 25 psi to create dull surfaces in order to reduce laser beam reflection during laser welding. The specimens were then cleaned using acetone solution to remove any oil residue. This procedure was left for 5 min as recommended by the American Welding Society. Finally, all specimens were dried with nitrogen gas to be ready for welding.

3.2.1.3.4 X- Ray checking

As titanium has high tendency to react with oxygen; casting of titanium in thin pieces may experience porosity. Therefore, x-ray images were obtained to check all the specimens to ensure that all specimens are free from porosity before the cutting and laser welding procedures start.

3.2.2 Cobalt-chromium(Co-Cr)

Thirty rectangular plastic patterns with the measurement according to the dental base metal alloys (ISO 6871-1) were prepared (1 thickness×3 width ×42 length mm). They were divided equally to 15 test specimens and 15 control specimens.

3.2.2.1 Cobalt-chromium Investing

Perform plastic patterns were invested in a mould with a phosphate bonded investment (type 2 dental stone). In this step, 100 g of investment powders (Wirovest, 51048, Bego, Germany) were mixed with 15mL of water. The mixture was spatulated and heavily vibrated under vacuum to eliminate the presence of trapped air and to produce a dense mix. The investment material was then poured carefully over and around the plastic models' surfaces. It was left for 45 min to allow it to set.

3.2.2.2 Cobalt-chromium casting technique

3.2.2.2.1 Heating cycle:

The mould and the crucible were placed in the oven at room temperature. The temperature was increased to 200^{o}C for 30 min to allow burnt out. The oven temperature was increased to 1000^{o}C for 30 min before casting.

3.2.2.2.2 Casting:

Casting started immediately after the heating cycle by using casting machine (Manfredi Neutrodyn Easyti, Italy). The mould and the crucible were placed in the casting machine chamber and fixed in the centre of fug arm. After that, casting procedure was started by towing up the fixed hand to allow the centrifuge of the melting cobalt-chromium to start. The melted alloy is then flowed it inside the mould until casting, and then the mould was allowed to cool down at room temperature.

3.2.2.2.3 Polishing

This procedure was same as mentioned for titanium polishing.

3.2.3 Welding

3.2.3.1 Preparation of specimens for laser welding:

Fifteen specimens cast Ti and 15 Co-Cr (1×3×42) mm were prepared. A perpendicular cut was made at the centre of each specimen. Square butt joint was used in this study to join Co-Cr and Ti. The square butt joint was used for metals that are small in thickness (Figure 3.9). Preparation of the joint is simple, since it only requires matching the edges of the plates together; however, as with any other joint, it is important that it is fitted together correctly for the entire length of the joint.

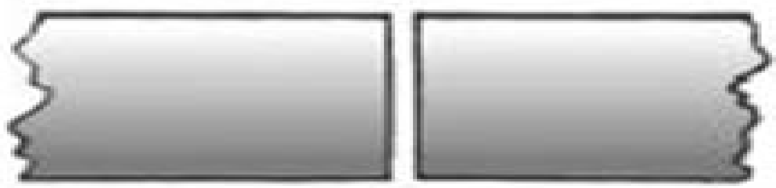

Figure 3.9 Square-butt joint

The cut surface of the 2 piece of sectioned specimen was finished using a grinding and polishing machine (Grinder/Polisher, Metaserv®, Buehler Ltd, Illinois, USA) as illustrated in Figure 3.10, with no 600 of silicon carbide paper.

Figure 3.10 Wheel grinding and polishing machine.

3.2.3.2 Laser welding

For laser welding of cobalt-chromium and titanium metal alloy joint surfaces (Co-Cr and CPTi), specimens were put against each other and placed in tight contact manually on the metal flex micrometric adjustable holder (Figure 3.11) to facilitate correct joint position) (Figure 3.12).

The laser welding apparatus (Manfredi jewellaser 50, Italy) (Figure 3.13) used in this study utilizes yttrium, aluminium and garnet crystals doped with neodymium (Nd:YAG laser) with a wavelength of 3000A was used with the following settings:

1. Power voltage of 270W.
2. Pulse duration of 10ms.
3. Welding spot diameter of 1.0mm.

These setting of laser conditions were chosen following the pilot. A pilot study was conducted to determine the right welding of the power and pulse duration. Three laser pulses (spot diameter: 1.0 mm x 3.0 mm width of specimens) were applied from both sides, ie., double welding technique was performed on both surface under Argon gas atmospheres to weld the entire joint width of the specimens. The Argon gas used to protect the molten metal oxidization and it does not cause any metallurgical problems.

Figure 3.11 Metal flex micrometric adjustable holder.

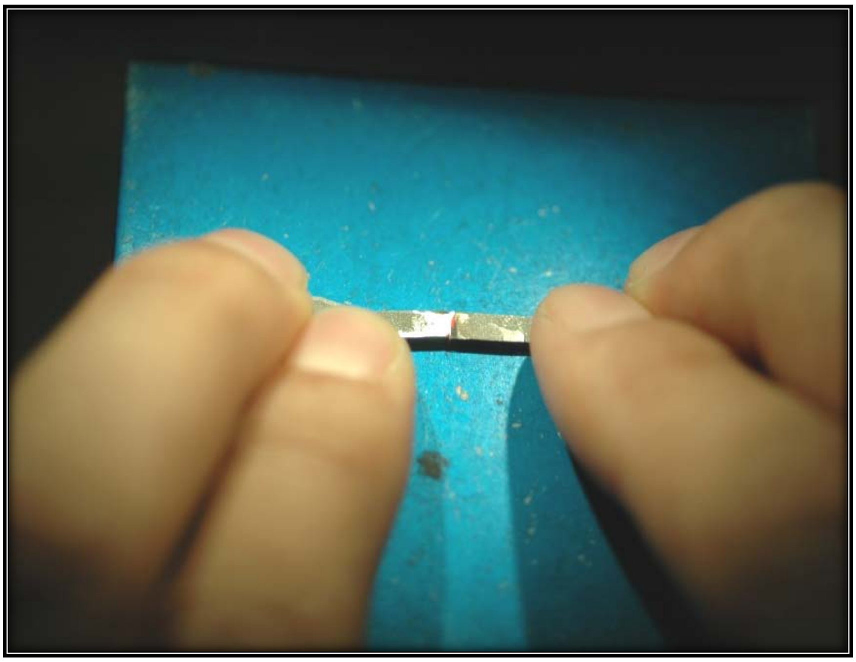

Figure 3.12 Specimens butted in tight contact manually

Figure 3.13: Laser welding machine.

3.2.4 Pilot study

Twelve specimens of titanium and cobalt-chromium were chosen, then they were divided into four (3 specimens) groups according to the welding laser power with increment of 30A between a group and another; 210A, 240A, 270A, and 300A respectively. The mean value of the tensile strength for each group was calculated (see Table 3.3)

The groups were:

(i) Group of 210A

(ii) Group of 240A

(iii) Group of 270 A

(iv) Group of 300 A

Table 3.3 Mean values of tensile strengths of the four groups of titanium-cobalt-chromium welded samples according to laser welding power.

Group No.	i	ii	iii	iv
Power/ A	210A	240A	270A	300A
Tensile strength	143.600 MPa	230.500 MPa	491.800 MPa	247.500 MPa

3.2.5 Tests

3.2.5.1 Tensile strength

Tensile strength test for specimens were carried out with the use of universal testing machine (Shimadzu Autograph AG-X, Japan) (Figure 3.14).

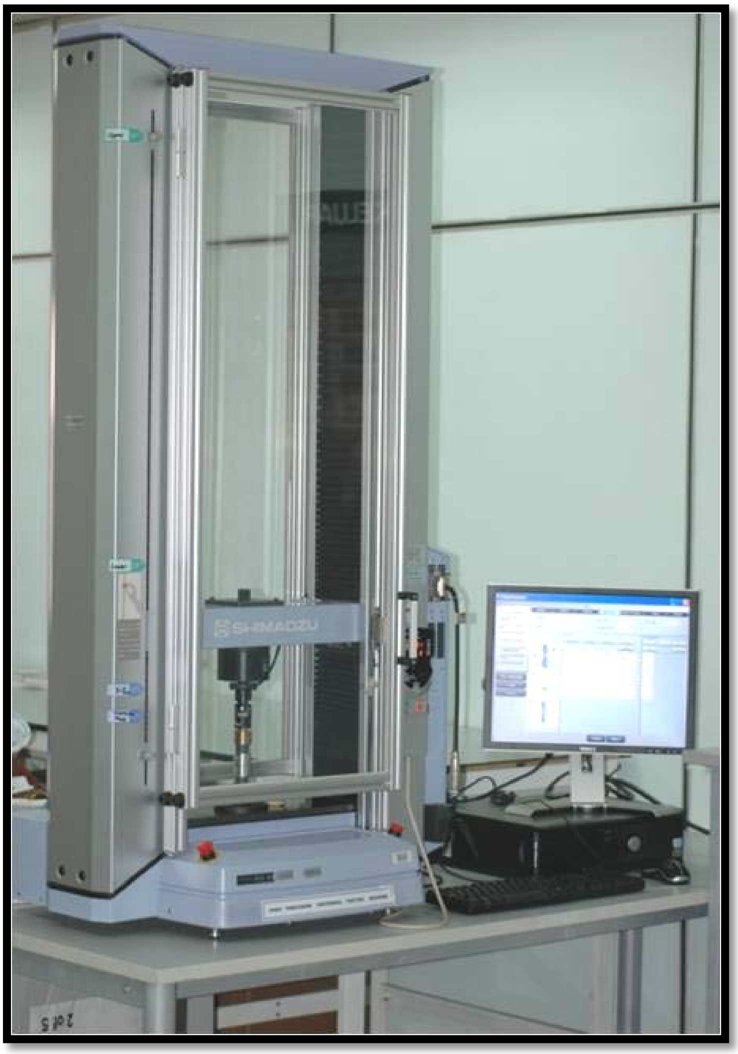

Figure 3.14 Universal testing machine (Shimadzu)

All specimens and the controls were carefully measured with digital callipers (Mitutoyo, Japan) before testing. The thickness and the width of each specimen were entered manually to the computer before specimen was placed on the joints. The specimens were then symmetrically placed at the centre of the machine and tensile test procedure was performed until the complete fracture of the specimen occurred. The test was performed at cross head speed of 0.5mm/min according to the ISO and gauge length of 20 mm.

3.2.5.2 Three-points bending test

Three-point bending test for the specimens were carried out using a universal testing machine (Shimadzu). The specimens were centred on the device in such a way that the loading wedge engaged the centre of the upper surface of the specimens. The distance between fulcrums was 20 mm and it was set up to travel at a crosshead speed of 0.5 mm/min according to the ISO. Specimens were loaded until fracture occurred. The three-point bending test was performed using the following formula:

$$\sigma_{u,b} = \frac{3Fl}{2bh^2}$$

where,

F is the load at failure, in Newton's;

L is the inner test span (centre to centre distance between inner support rollers), in millimetres;

b is the width of the specimen, i.e., the dimension of the side at right angles to the direction of the applied load, in millimetres;

h is the thickness of the specimens, i.e., the dimension of the side parallel to the direction of the applied load, in millimetres.

3.2.5.3 Modulus of elasticity.

Modulus of elasticity test was measured with the tensile strength test. Modulus of elasticity testing was conducted with a universal testing machine (Shimadzu) at a crosshead speed of 0.5 mm/minute and a gauge length of 20 mm (grips were attached 5 mm from both ends). The breaking stress (MPa) was recorded when the specimen fractured, and the means and standard deviations were calculated (n=5).

3.2.5.4 Microscopic examination for microstructure

Two specimens from titanium and cobalt-chromium group were randomly selected for examination under Scanning Electron Microscopy (SEM) with low vacuum operating mode(Quanta 200 F, FEI, Hillsboro, USA) (Figure 3.15). The backscatter electron (BEI) mode was used to investigate the topography of the specimen under laser welding.

Figure 3.15 Scanning electron microscopy.

3.2.6 Statistical analysis

The data was analysed using SPSS 16for windows. Normality was tested then Mann Witney(U) test and *t*-test were conducted to verify the difference between the study group and control group at (α=0.05).