CHAPTER 1

Introduction And Literature Review

1.1 BIOMATERIALS

A biomaterial is a non-viable material used in medical devices to test, treat, enhance, or substitute any tissue, organ, or function of the human body [1]. Biomaterial performance and applicability in biological systems are crucial for the development of biomedical implants and tissue engineering. Metallic materials such as stainless steel, cobalt alloys, titanium alloys, ceramics like aluminium oxide, zirconia, calcium phosphates, as well synthetic and natural polymers, can be employed in human body. Titanium (Ti) and titanium alloys are among the most important biomaterials due to their resistance to body fluid effects, tensile properties, flexibility, and high resistance to corrosion. The combination of strength and biocompatibility make them attractive for medical applications. For example, commercially pure Ti (Cp-Ti) is one of the most often used material for dental implants, whereas Ti-6Al-4V alloy is chosen for orthopaedic applications [2].

Various methods of surface modification of titanium and its alloys are discussed in this chapter, including the promising method of developing surface nano crystallization such as ultrasonic shot peening. It also includes the most recent research related to evaluation and use of this process along with importance of this process of surface modification for Ti and its alloys for biomedical applications and future perspectives.

1.2 TITANIUM AND TITANIUM ALLOYS

Titanium is a hard, shiny, light metal with a high strength-to-density ratio, with yield strength of some of its alloys ranging from 470 to 1060 MPa, depending upon its grade. There are two allotropic forms of titanium; alpha(α) Ti with hexagonal-close-packed (HCP) crystal structure, that transforms to beta (β) Ti, body-centered-cubic (BCC) crystal structure, at the transition temperature of 882°C. Like some other metals, transition temperature of titanium varies with alloying elements [3,4]. Commercially pure titanium has high corrosion resistance and is biocompatible with the human body due to its ability to generate an inert oxide layer on its surface.

Ti alloys are essential materials for design engineers because of their high mechanical properties and high corrosion resistance in hostile environments, with densities ranging from 4.43 g/cc to 4.85 g/cc depending on alloying elements. Ti alloys are biomaterials superior to other competing alloys such as SS 316L and Co-Cr alloys, due to their unique combination of mechanical properties, low densities, non-toxicity, and higher biocompatibility [5].

1.2.1 Classification of titanium alloys

Alloying elements significantly affect the crystalline structure of titanium and different types of alloys are formed. The following classifications of Ti alloys have been established: alpha (α), alpha + beta (α+β), and beta (β) alloys. Figure 1.1(a) shows crystal structure of the α (hcp) and β (bcc) titanium and Figure 1.1(b) shows categories of titanium phase diagrams. While some alloying elements like Sn and Zr remain neutral and do not affect the stability of the α / β phases, alloying elements Al, O, N, C are α stabilizers and increase the α phase field. Oxygen and nitrogen are highly soluble in titanium and powerful α stabilizers, allowing the lower temperature α phase to emerge immediately from the liquid as an intermediate disordered phase. On the other hand, there are several alloying elements, as shown in Figure 1.1(b), which are β stabilisers; among these Mo, V, Ta, Nb are β isomorphous and Fe, Mn, Cr, Co, Ni, Cu, Si, H are β eutectoid type β stabilizers.

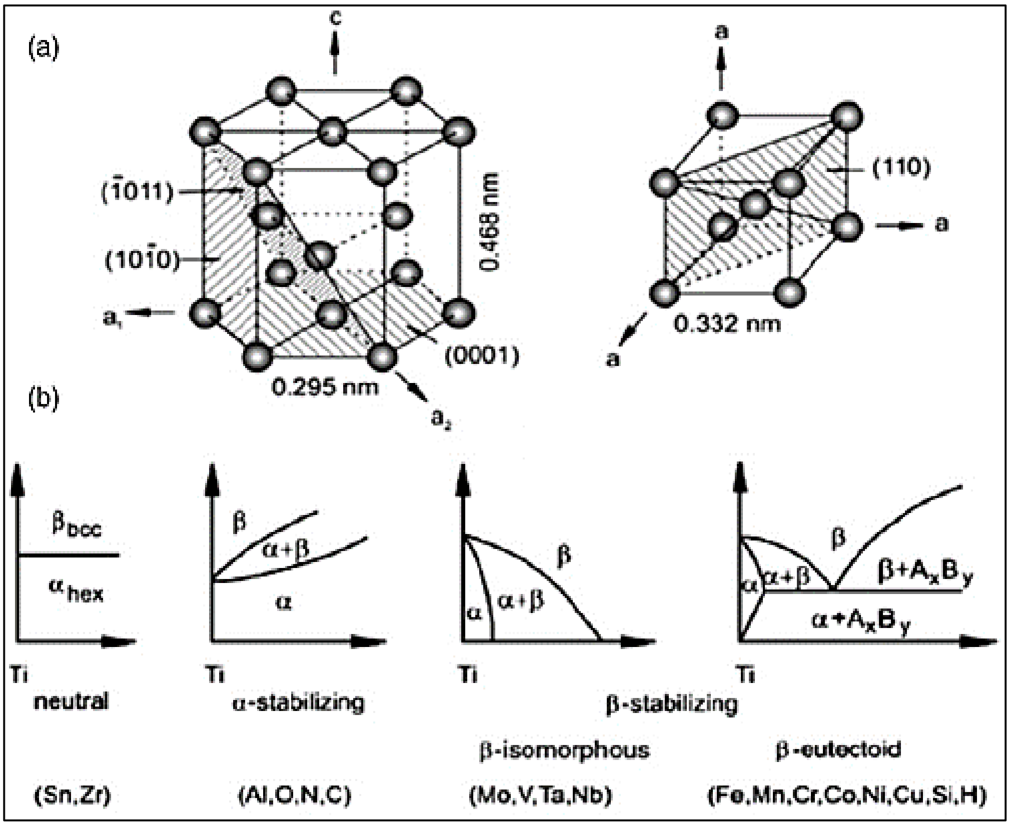

Figure 1.1 (a) The hcp (alpha) and bcc (beta) structure of titanium. (b) Categories of titanium phase diagrams formed with different alloying additions.

Mo and V refractory metals generate an immiscibility gap in the β phase, resulting in a monotectoid reaction, whilst other stabilizers such as Cr, Fe, Cu, Ni, and Si form intermetallic compounds via eutectoid reaction [4].

1.2.1.1 Alpha titanium alloys

These alloys have yield strengths and elastic moduli ranging from 170-485 MPa and 102-104 GPa, respectively, and ultimate tensile strengths ranging from 240–550 MPa (see Table 1.1). Rather than heat treatment, structural changes are typically produced by the proper use of alloying elements. They are formable, weldable and have lower strengths than other Ti alloys. To boost strength, they often contain alpha stabilisers such as aluminium, germanium, or carbon, while some comprise beta stabilisers such as molybdenum, vanadium, or niobium. Some α, α+β, β titanium alloys [6,7], with their tensile properties are presented in Table 1.1.

Table 1.1 Some α, α+β, β titanium alloys and their tensile properties [8].

Alloy	Tensile strength (UTS) (Mpa)	Yield strength (σy)	Elongation (%)	RA (%)	Modulus (GPa)	Type of alloy
1. Pure Ti grade 1	240	170	24	30	102.7	α
2. Pure Ti grade2	345	275	20	30	102.7	α
3. Pure Ti grade 3	450	380	18	30	103.4	α
4. Pure Ti grade 4	550	485	15	25	104.1	α
5. Ti–6Al–4V ELI (mill Annealed)	860–965	795–875	10–15	25–47	101–110	α + β
6. Ti–6Al–4V (annealed)	895–930	825–869	6–10	20–25	110–114	α + β
7. Ti–6Al–7Nb	900–1050	880–950	8.1–15	25–45	114	α + β
8. Ti–5Al–2.5Fe	1020	895	15	35	112	α + β
9. Ti–5Al–1.5B	925–1080	820–930	15–17.0	36–45	110	α + β
10. Ti–15Sn–4Nb–2Ta–0.2Pd						
(Annealed)	860	790	21	64	89	
(Aged)	1109	1020	10	39	103	
11. Ti–15Zr–4Nb–4Ta–0.2Pd						α + β
(Annealed)	715	693	28	67	94	
(Aged)	919	806	18	72	99	
12. Ti–13Nb–13Zr (aged)	973–1037	836–908	10–16	27–53	79–84	β
13. TMZF (Ti–12Mo–6Zr–2Fe) (annealed)	1060–1100	1000–1060	18–22	64–73	74–85	β
14. Ti–15Mo (annealed)	874	544	21	82	78	β
15. Tiadyne 1610 (aged)	851	736	10		81	β
16. Ti–15Mo–5Zr–3Al						β
2003(ST)	852	838	25	48	80	
(aged)	1060–1100	1000–1060	18–22	64–73		
17. 21RX (annealed) (Ti–15Mo–2.8Nb–0.2Si)	979–999	945–987	16–18	60	83	β
18. Ti–35.3Nb–5.1Ta–7.1Zr	596.7	547.1	19.0	68.0	55.0	β
19. Ti–29Nb–13Ta–4.6Zr (aged)	911	864	13.2		80	β

1.2.1.2 Alpha + Beta titanium alloys

These alloys contain both α and β stabilising alloying elements and their mechanical properties depend on their chemical compositions. They have a wide range of mechanical properties, such as tensile yield strength (795-1020 MPa), elastic modulus (89-114 GPa), and UTS (715-1109 MPa) (Table 1.1), as well as moderate creep resistance and may be suitably adjusted by alloying and heat treatment. Thermal processes affect their properties to a large extent by varying the amount and distribution of the β phase.

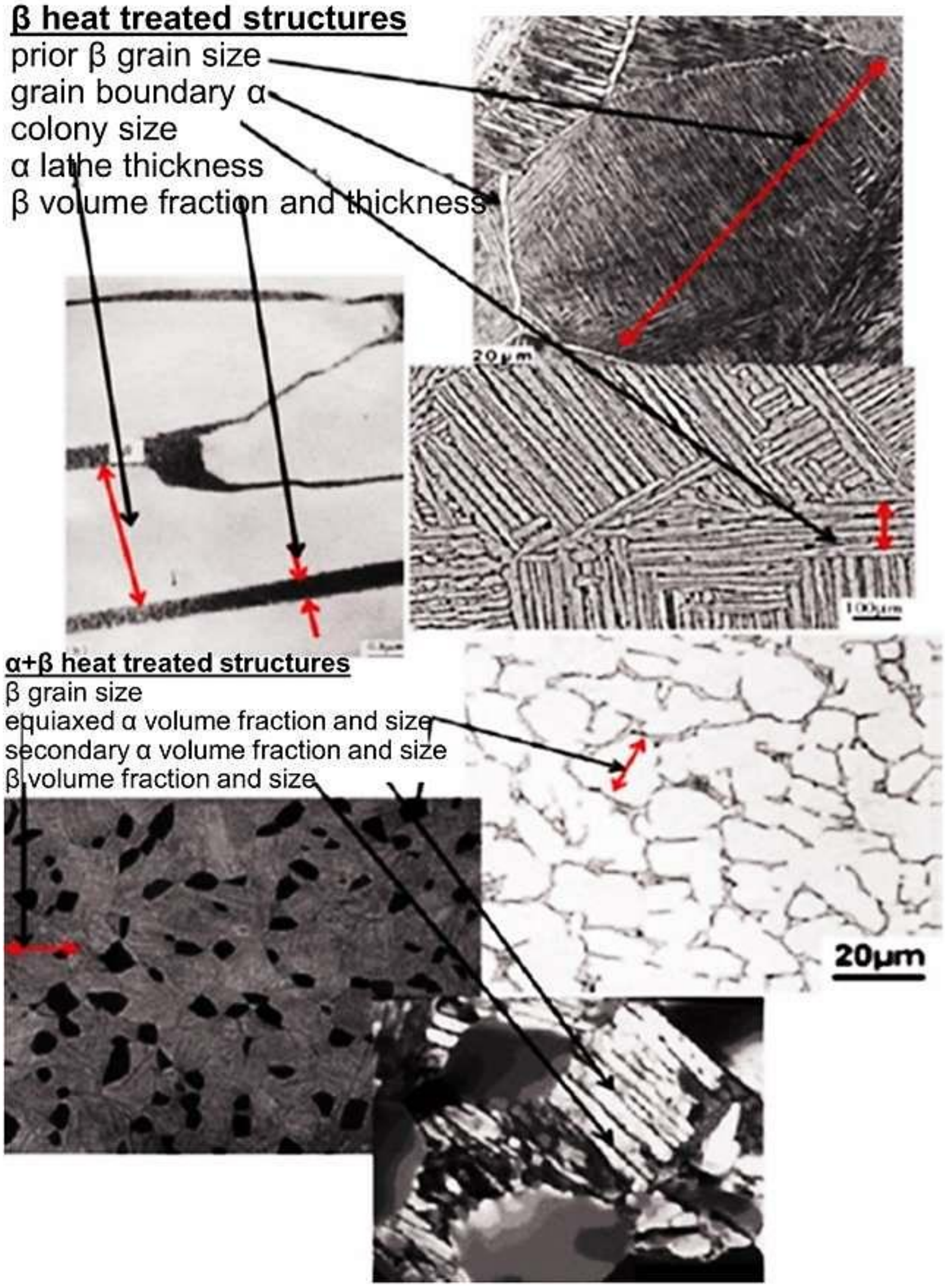

Figure 1.2 Key microstructural phases observed in various types of microstructures in a wide range of titanium alloys [9].

Ti alloys develop mainly equiaxed α-grains and acicular martensite (Figure 1.2) when quenched within the α+β temperature range, and can also be strengthened by ageing treatment [10]. In the heat-treated condition, they are frequently employed for high-strength applications and undergo annealing at temperatures ranging from 705-845°C, while solution treatment temperatures range from 900-955°C, followed by quenching and ageing at 480-593°C for 2-24 hrs. Because of its high mechanical properties, corrosion resistance, and biocompatibility, the α+β titanium alloy (Ti-6Al-4V) is commonly used for medical implants. As a biomaterial, the extra low interstitials (ELI) grade alloy, which contains minimal levels of interstitial elements, is employed. The Ti-6Al-4V ELI alloy has high toughness and is utilized for bone fixation plates and prosthetic hip joint stems [11]. In Ti-6Al-4V alloy, impurities reduce fatigue strength through notch effect. Nonetheless, there are worries about the harmful effect of Ti-6Al-4V alloy ions produced on tissues surrounding the implant; since vanadium has possible cytotoxicity and unfavourable tissue interactions, aluminium ions on the other hand cause long-term Alzheimer's disease. To support this effect, Morais et al. [12] evaluated Ti alloy implants in rabbit tibiae and examined the effect of vanadium ion released while the implant healed after 1, 4, and 12 weeks. The lungs, liver, and kidneys were taken and tested; the results showed a minor rise in vanadium content after the first week, and a large increase during a four-week period, with a slight drop after 12 weeks. Wilke et al. [12] on the other hand claimed that long-term interaction of human bone marrow cells with Ti-6Al-4V alloy particles causes large release of pro-inflammatory and osteolytic mediators, which aids implant loosening and revision surgery complications. As a result, Ti alloys with reduced toxicity have been developed by substituting Nb, Ta, Zr, and hafnium (Hf) for V and Al components. Ti-6Al-2.5Fe, Ti-6Al-2Nb-1Ta, Ti-6Al-7Nb, and Ti-15Zr-4Nb-4Ta alloys are among the other α+β Ti alloys produced [13,14].

1.2.1.3 Beta titanium alloys

These alloys have yield and tensile strengths of 545-1060 MPa and 850-1100 MPa, respectively, which may be obtained by cold work, alloying, and ageing treatments. They are metastable, have high strengths but little ductility when aged. They have a low elastic modulus (55-85 GPa), poor stability at 200-300°C, little creep resistance, and cannot be welded without embrittlement. These alloys are annealed and solution treated at temperatures ranging from 730 to 980°C, and are aged for 2–48 h at temperatures ranging from 482 to 593°C for the best combination of mechanical properties. Duplex ageing is usually performed on these alloys to further improve their qualities. The first ageing treatment is performed between 315°C and 455°C for 2-8 h, and the second ageing treatment is performed between 480°C and 595°C for 8-16 h [4]. Another issue with (α+β) titanium alloys is their high modulus (110 GPa) in comparison with that of human bone (20-30 GPa). High stiffness causes a stress shielding effect, and bone resorption. Due to this, metastable Ti alloys with low modulus have been developed which reduce the stress shielding effect during bone fixation. The developed β-Ti alloys are: Ti-15Mo [15], Ti-15Mo-5Zr-2Al, Ti-13Nb-13[16], Ti-35Nb-7Zr-5Ta [17], Ti-29Nb-13Ta-4.6Zr [18], Ti-15Mo-3Nb-0.3O (21SRx) [6], Ti-Nb-Zr-Ta alloys (TNZT) [18], Ti-35Nb-2Ta-3Zr [19], Ti-24Nb-4Zr-7.5Sn [20], Ti-36Nb-5Zr and Ti-8Nb-28Zr [21].

1.3 BIOMEDICAL APPLICATIONS OF TITANIUM ALLOYS

The shoulder, elbow, wrist, hand, as well as the hip and knee joints, are all examples of articulations in the human skeletal framework. The hip and shoulder joints are known as ball-and-socket joints because they enable free bending and rotation. A hinge joint is also used to describe the elbow joint, which can only move in one direction, whereas the oval joint is used for the wrist joint, which can move in both directions [22]. Ti alloys are used in a variety of biomedical applications, including spinal fixtures, hip and knee prostheses, orthopaedic implant finger plates, ankle replacements, and oral prostheses [23] (see Figure 1.3).

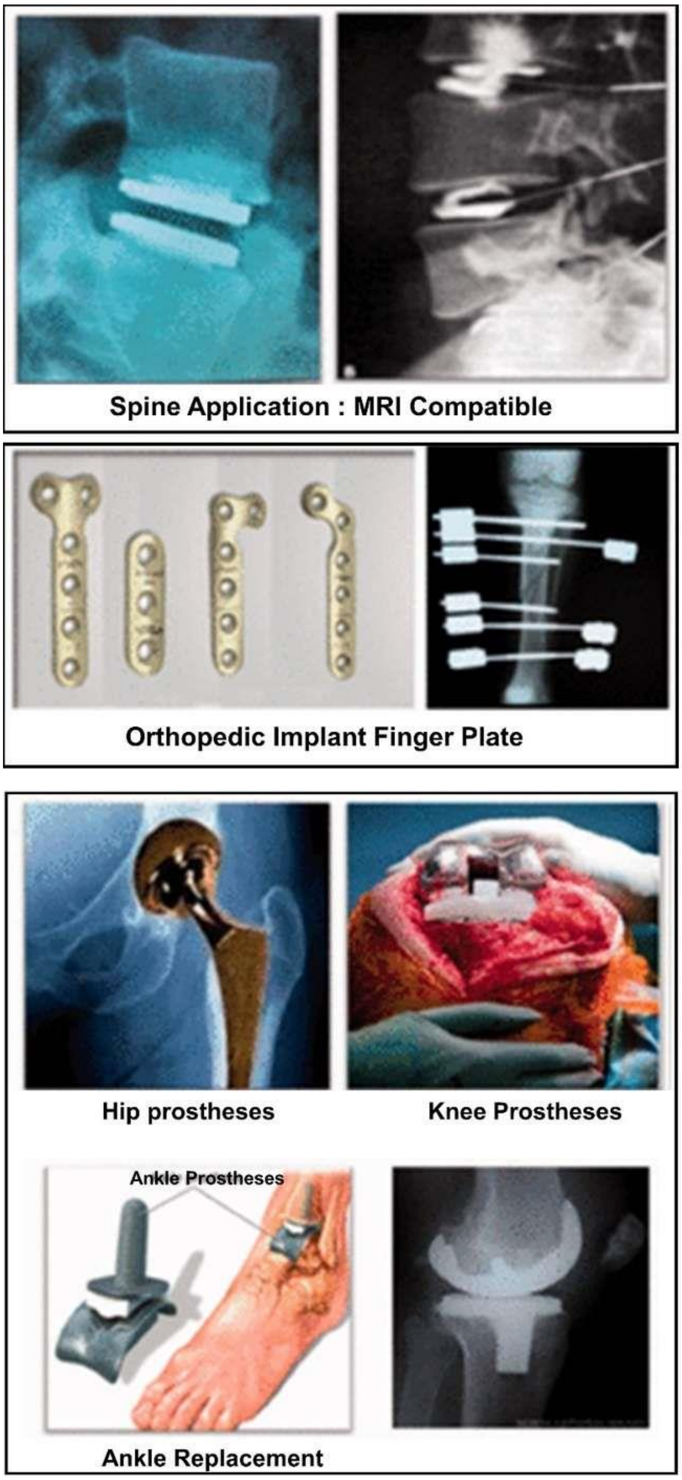

Figure 1.3 Biomedical applications of titanium alloys [23].

Apatite and collagen fibres with anisotropic features make up human bone tissue, which is a composite type of material. The highest strength and flexibility of cortical bone are found in the bone axis direction, whereas these are lowest in the transverse direction of the bone axis, giving bone tissue considerable compression resistance but not tension or torsion resistance. When maximal loads are applied to bones in the arm or legs, they break, but during osteoporosis, they fracture easily. Implants made of stainless steel or titanium alloys are commonly utilised to heal such fractures. Ti alloys, on the other hand, have greater corrosion resistance and biocompatibility, making them preferable to stainless steel. Bone also contains chemical and inorganic components that provide flexibility as well as strength and toughness. The organic components of bone include collagen, fibrillin, glycoproteins, and proteoglycans, whereas the inorganic component is hydroxyapatite. Compact bone, also known as cortical bone, and trabecular bone, sometimes known as cancellous bone, are two primary types of bone. The anisotropic and porous trabecular bone has lower strength and stiffness than compact bone. Bone remodelling is a self-healing process that is essential for bone restoration. The dynamic remodelling process of bone homeostasis is characterised by early osteoclasts, i.e., bone tissue resorption, followed by new bone formation (osteoblasts) [24].

The load transfer characteristics of a joint are the fundamental prerequisite for the design of its replacement in order to minimise bone resorption and ultimate loosening caused by overloading of the implant-bone contact or insufficient load transfer. Similarly, inert behaviour of titanium promotes implant encapsulation by the body. Furthermore, implant loosening may occur as a result of the micro-movements of implant, opposing direct bonding to the bone [25]. The causes of greater implant replacement include, first and foremost, the continual ageing population, with an estimated 90% of the population over the age of 40 suffering from degenerative illnesses, as well as a rising elderly population. According to records, 30% of persons in need of hip replacements are under 65 years age, and with a standard average life of implants 12-15 years, a higher number of patients will require revision surgery at some point throughout their lives. As a result, the recent considerable increase in revision surgeries is due to the degenerative illnesses such as osteoporosis, osteoarthritis, and trauma inducing bone joint disintegration in humans [26].

1.4 IMPACT OF MICROSTRUCTURAL MODIFICATION ON MECHANICAL PROPERTIES

The majority of metallic biomaterials have crystalline structures, with dislocation and twinning influencing their mechanical properties. As a result, careful regulation of lattice defects and phase changes improves mechanical characteristics. Furthermore, structural management of metallic biomaterials by plastic deformation and heat treatment increases their mechanical performance to fit a specific application. Figure 1.2 depicts the key microstructural phases seen in a wide range of Ti alloys. There are many microstructural length scales associated with the α phase, ranging from very fine α laths in β-enriched alloys to colonies of similarly orientated laths and the equiaxed morphology. Beta grain sizes vary greatly, ranging from several hundred microns in β heat-treated near α-alloys to tens of microns in α+β processed alloys [27].

Table 1.2 Tensile properties of selected metallic biomaterials in annealed condition.

Metallic Biomaterials	Tensile Properties		
	Elastic Modulus (GPa)	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)
CoNiCrMo	232	241	793
Cp-Ti, grade 4	110	485	550
316L Stainless steel	193	190	490
Ti-6Al-4V	116	795	860

Microstructure dependent mechanical parameters include fracture toughness, fatigue strength, ductility, ultimate strength, and yield strength. The tensile mechanical properties of the major metallic biomaterials are provided in Table 1.2. When compared to the other metallic biomaterials on the list, Ti-6Al-4V alloy has the low elastic modulus (116 GPa), the highest yield strength, and the highest ultimate tensile strength (795 MPa) and (860 MPa), respectively. Ti alloys are used in biomedical applications because of their unique mix of optimal mechanical properties. According to this table, the yield strength of biomedical Ti alloys is between 500 and 1000 MPa, their elongation is between 10 and 20%, and their fatigue strength is between 265 and 816 MPa [28]. Lattice defects, such as point and planar defects, and their interactions with dislocations give rise to these properties. Metallic biomaterials are typically used in load-bearing applications, mostly to replace hard tissues, and hence must have high fatigue strength and fracture toughness to sustain the daily requirements of human activities [29]. The capacity of metallic biomaterials to go through plastic deformation causes acute defects to be blunted, resulting in lower local stress conditions and higher fracture toughness [23]. Furthermore, Ti alloys have a modest elasticity, allowing for equal load distribution between implants and bone. The elastic modulus of β-type Ti alloys (55-85 GPa) is lower than that of α alloys (102.7-104.1 GPa) and α+β (90-114 GPa) types Ti alloys, as well as other metallic biomaterials such as SS (206 GPa) [24] and Co alloys (240 GPa), as indicated in Table 1.2. Despite the fact that some newly developed β-Ti alloys have a modulus somewhat greater than bone with appropriate alloying elements, their wear resistance and strength under loading circumstances are unsatisfactory [26]. In total hip replacements, fracture of the femoral stem formed of Ti alloys has not been common throughout the years. However, a rise in the number of hip replacement surgeries linked to modular junctions has lately been recorded. The stem-taper junction was the site of the majority of the fatigue fractures [30,31]. Grupp et al. [32] looked over 5000 examples of modular Ti femoral neck implants. Within the first two years of implantation, fatigue fracture was found in up to 1.4 percent of them, according to the study. As previously stated, the mechanical characteristics of metallic biomaterials are affected by both the microstructure and the crystallographic texture. Texture controls the availability of densely packed crystallographic planes and the resulting surface energy, which increases corrosion resistance, osteoblast proliferation, and surface osteogenesis. The distribution of various orientations of the constituent grains inside a material is referred to as its polycrystalline texture. The basal pole orientations also have an impact on the surface energy of a material's interface. Surface energy differences, the grain orientation of Ti-6Al-4V alloy influenced its osteoblast interactions [33]. Hoseini et al. [34], on the other hand, proposed that cell adhesion to Ti surfaces is influenced by texture rather than grain size. Cell attachment and proliferation have also been shown to be influenced by surface wettability, which is mediated by protein adsorption [35].

Nanostructured metals are clearly promising biomaterials for orthopaedic applications since they increase mechanical properties while also promote osteoblast adhesion and proliferation, extending implant life. Even in the absence of soluble osteogenic stimuli, Dalby et al. [36] found that nanopatterned Ti causes mesenchymal progenitor cells to deposit bone mineral by altering protein adsorption and cytoskeletal architecture. Metals undergo severe plastic deformation (SPD), which results in nanocrystallization [37]. However, SPD method has certain apparent drawbacks, such as cost of expensive equipment, poor throughput, and the need for specialised expertise [23]. As a result, when it comes to forming nanocrystalline surfaces on metallic substrates, the surface mechanical attrition treatment (SMAT) approach outperforms the

SPD technique [38]. The SMAT method is suitable for high-strength materials such as stainless steel and Ti alloys.

Initially, commercially pure titanium (Cp Ti) was employed as an alternative to stainless steel and Co-Cr alloys [39–41]. However, there were three key issues with Cp Ti: (a) the modulus of elasticity of Cp Ti was high (110 GPa) in comparison to human bone, (b) substantial wear loss, and (c) weak mechanical properties in comparison to hard tissues. To address these issues, Cp Ti was substituted by α+β Ti-based alloys, particularly Ti-6Al-4V alloy. Cp Ti and α+β type Ti alloys were initially meant to be utilized as general structural materials, particularly for aircraft structures, and were only later employed for biomedical purposes.

In recent times many titanium alloys have been developed. The Ti–6Al–4V alloy became the accepted standard, and it was the first titanium alloy used in biomedical applications. However, current research shows that Al and V can have hazardous effects and have a higher elasticity modulus (about 120 GPa) than bone (around 28 GPa). The likelihood of V and Al emission, as well as the rising usage of prostheses, has prompted the creation of novel biomedical alloys free of harmful metals.

V free α+β Ti alloys such as Ti-6Al-7Nb and Ti-5Al-2.5Fe [28] were developed to minimize the toxicity of V by substituting V with Nb and Fe. When compared to the Ti-6Al-4V alloy, both of these alloys have superior mechanical characteristics. However, the modulus of elasticity of certain α and α+β alloys is significantly greater [16-18] than that of human bone, resulting in a stress shielding effect [42,43]. Therefore, low modulus β titanium alloys [44] containing stabilizing elements such as Ta, Nb, Zr, and Sn have been produced because of their non-toxicity [45], increased mechanical properties, and better tissue response [46,47]. Several characteristics must be assessed to establish acceptability of a material for body implant applications. Corrosion behaviour is particularly important among these characteristics, because ion leakage from the implant into the surrounding tissues might create biocompatibility issues [48]. Corrosion resistance of biocompatible alloys used as orthopaedic implants is critical, not only because it defines the useful life of device, but also because corrosion processes in live organisms can be hazardous. Corrosion products have been shown to have an impact on cell metabolism [33]. Ti alloys containing non-toxic elements may have a lower elastic modulus than Ti–6Al–4V. Alloys with elements such as Nb, Zr, Ta, Pt, and Ti have been thoroughly investigated, as these are the only five elements known to have no adverse tissue reaction [49]. The elastic modulus of titanium alloys is well known to be a critical factor for implant applications. Long-term tests, on the other hand, show that the high moduli of titanium implants transfer insufficient load to nearby remodeling bone, resulting in bone resorption and ultimate loosening of the prosthetic devices [50].

Davidson and Kovacs [51] developed the Ti-13Nb-13Zr alloy, which is part of a novel family of biomedical alloys for application as medical implants. It is a near β-Ti alloy consisting of hcp martensite in water-quenched condition. The aged alloy is composed of hcp martensite and submicroscopic bcc β precipitates. The material is strengthened and hardened by the distributed beta precipitates. For titanium alloys, ageing treatment usually leads to the conversion of martensite into a mixture of α, β and α´´phases. The presence of martensite in the aged alloy suggests that it is a more α alloy rather than a near-β alloy. This alloy has modulus of elasticity around 65 GPa in its aged condition. Nb strengthens the alloy through solid solution strengthening, as well as lowers the elastic modulus and transition temperature [52]. The Nb content of the material is essential because as the Nb concentration increases, the fraction of α phase decreases. The addition of Zr to Ti improves the strength of the alloy and elastic modulus [53].

1.4.1 Surface Modification Techniques

Various surface modification procedures, such as laser shock peening, conventional shot peening, ultrasonic shot peening, and high-energy shot peening, have enhanced the surface attributes of structural components. By delaying the process of crack initiation and initial crack propagation, these techniques could be extremely effective in improving fatigue life of structural components due to the combined effect of surface grain refinement and the associated compressive residual stresses in the surface and subsurface regions.

Among the several surface modification methods, ultrasonic shot peening is a relatively novel method of producing surface nanostructure. It has a considerable potential for enhancing mechanical properties of metal and alloys, without affecting their chemical composition.

1.4.1.1 Laser Shock Peening

A surface engineering method known as laser shock peening or laser peening is used to increase fatigue strength and corrosion resistance. The compressive residual stress created by laser peening enhances the resilience of materials to surface-related failures including fatigue and corrosion. In this process, a high-energy pulsed laser beam strikes the metal component, causing high-amplitude stress waves. Figure 1.4 depicts the laser shock peening process parameters. The surface of material resists stretching caused by stress waves and that results in creation of a compressed membrane. In laser peening, a laser wavelength of around 1 μm, a pulse duration of 10-50 nanoseconds, a pulse energy of 50-100 joules, and a beam width of 5 mm are utilized.

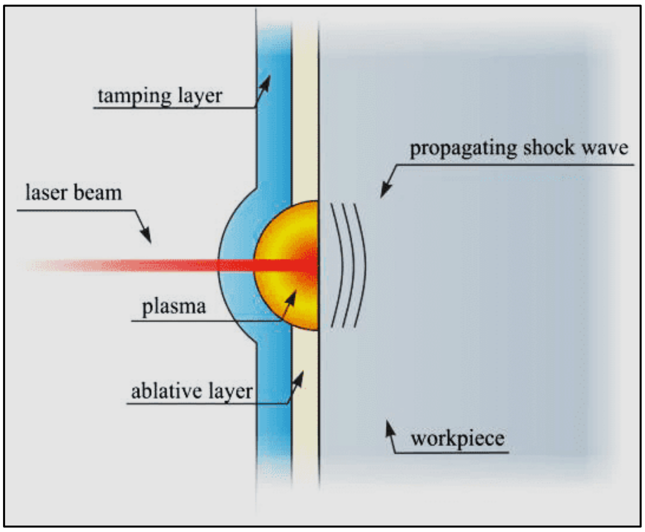

Figure 1.4 Laser shock peening process[54].

In respect of shot peening, compressive residual stress generated by laser shock peening is up to 4 to 5 times deeper in materials therefore the process of crack initiation is delayed and also, the rate of crack propagation is reduced under cyclic loading by the compressive residual stresses. A number of alloys used in aviation engines, airframes, and other technical applications have been subjected to laser shock peening. It was successfully applied on the fans and compressor blades of aero-engines to increase their damage tolerance.

1.4.1.2 Shot Peening

Shot peening (SP) is a cold working process that improves mechanical characteristics of metals, alloys, and composites through grain refinement in conjunction with compressive residual stress. Figure 1.5 shows the process of conventional shot peening schematically. In this method, small spherical shot media (metallic, glass, or ceramic) of proper hardness are accelerated in various types of peening devices and hit the workpiece surface.

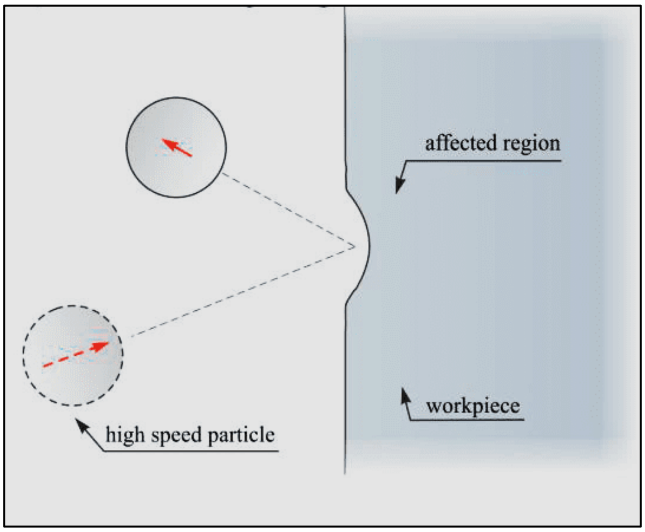

Figure 1.5 Shot peening process[54].

The main advantage of shot peening is delay in fracture development in highly tensile stressed components owing to grain refinement and compressive residual stresses near the surface. These effects are extremely beneficial in reducing corrosion and fatigue failure of materials. According to Umemoto et al. [55], air blast shot peening (ABSP) is the most common method of shot peening. ABSP is a shot peening technique in which compressed air is used to deliver shots onto the component through a nozzle. The shot velocity is kept above 100 m/s in this method, and the shot to specimen contact direction is almost perpendicular [56].

1.4.1.3 Ultrasonic Shot Peening

The ultrasonic shot peening (USSP) technique, also known as surface mechanical attrition treatment (SMAT), works on the principle similar to shot peening. Table 1.3 displays key differences between the conventional shot peening and USSP. USSP is a room-temperature cold working process in which the workpiece surface is exposed to impact of tiny balls vibrating at ultrasonic frequency, causing severe plastic deformation.

The balls collide with the surface to be peened. As demonstrated in Figure 1.6, steel balls striking the material surface produce dimples and compressive residual stress.

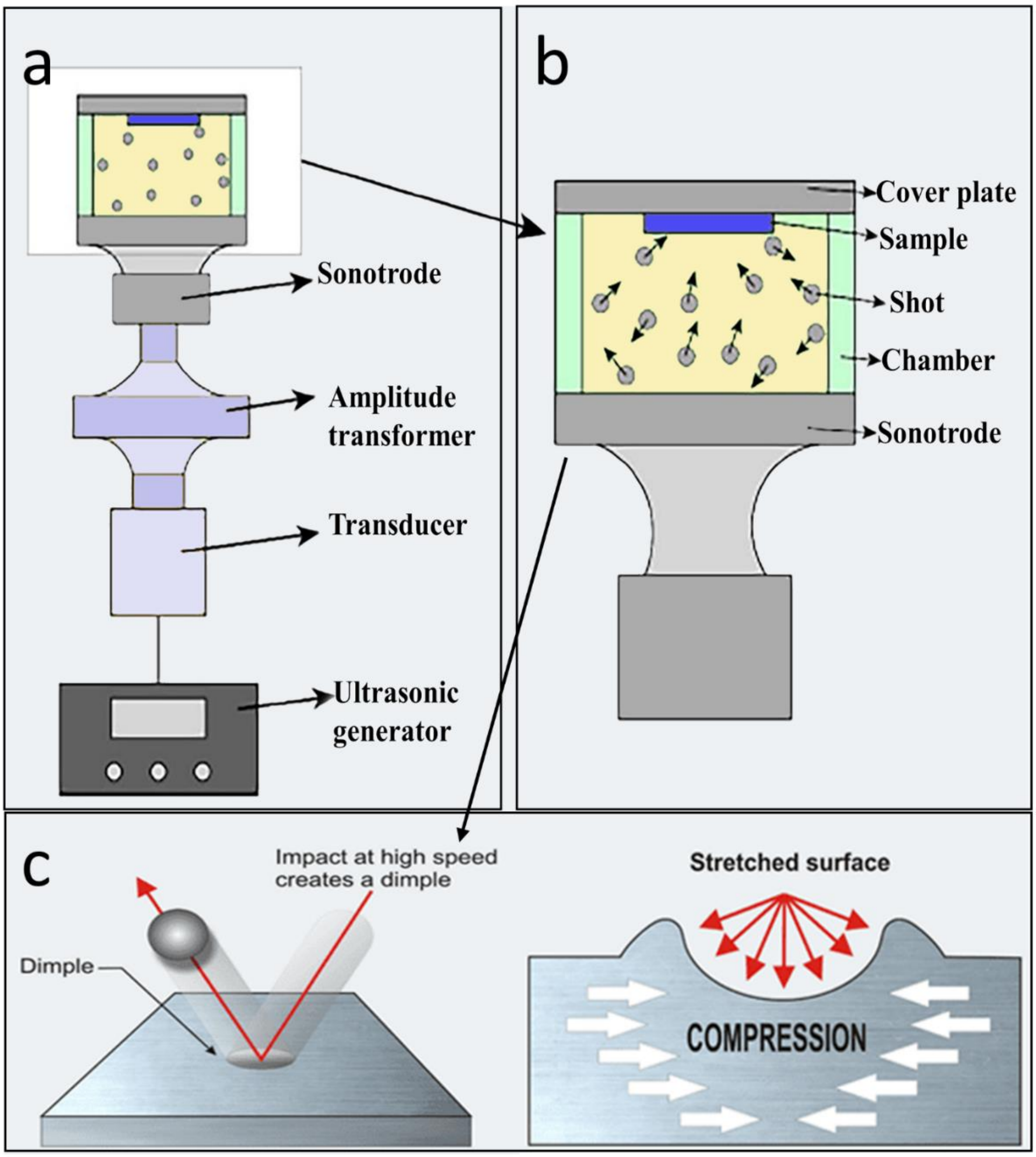

Figure 1.6 Schematic of (a) ultrasonic shot peening instrument, (b) chamber assembly, and (c) principle of ultrasonic shot peening [57,58].

This process modifies microstructures in the surface region, ranging from sub-micron to nano size. As compared to traditional shot peening, USSP produces less roughness and smoother surface quality. Treatment time, mass of the balls in the housing, and amplitude of sonotrode vibration, are the relevant parameters to be controlled in order to prevent the crack initiation from the impacts of shots on the shotpeened surface. These settings can be computer-controlled for the repeatability. USSP comprises an acoustic assembly, which includes a piezoelectric transducer, a booster, and a sonotrode as shown in Figure 1.6a. The acoustic assembly shown in Figure 1.6b generates mechanical vibrations, which are then transmitted to move steel shots. A piezoelectric transducer produces ultrasonic waves at a frequency of 20 kHz, which are amplified as they pass through an acoustic booster and into a chamber containing the sample.

Table 1.3 The basic difference between SP and USSP [59].

S. No.	**Parameters**	**SP**	**USSP**
1.	Balls size	0.25 mm to 1 mm	1 mm to 8 mm
2.	Velocity	20 m/s to 150 m/s	3 m/s to 20 m/s
3.	Frequency	50-200 Hz	20 kHz
4.	Roughness	Higher	Lower
5.	Compressive residual stress	Lower depth of compressive stresses	Higher depth of compressive stresses

1.4.1.4 High Energy Shot Peening

Another approach for generating nanostructured surface layers is high-energy shot peening. The high-energy shot peening treatment works in a way similar to surface mechanical attrition but with fewer and larger shots. The whole surface of the material to be treated is peened by high-energy flying shots, and a nanocrystalline layer is created [60]. By forcing strong plastic stresses onto the surface of metals and alloys, high-energy shot peening is one of the most successful processes for the fabrication of diverse ultrafine-grained structures. The plastic strain in the matrix has a gradient distribution in the surface layer, with a maximum at the top surface and a near-zero distribution farther into the matrix. Microstructure observation at various depth levels can thus give insight into the structural development process relevant to different phases of plastic strain [61].

1.5 IMPORTANCE OF ELASTIC MODULUS

During the last decade, there has been extensive usage of Cp -Ti and Ti-6Al-4V alloy for biomedical implants. Different alloys were developed for aerospace applications, among them, Ti-6Al-4V was the first titanium alloy to be used as biomedical implants [62,63]. Titanium and its alloys have lower modulus of elasticity than other metallic biomaterials such as stainless steels and Co-Cr alloys [62]. In general, elastic moduli of β-type titanium alloys is much lower than those of α and (α + β) type titanium alloys [64]. However, some health issues such as Alzheimer's neural disorder and Osteomalacia have been reported to be associated with the release of vanadium and aluminium ions from (α + β) type Ti-6Al-4V alloy [65].

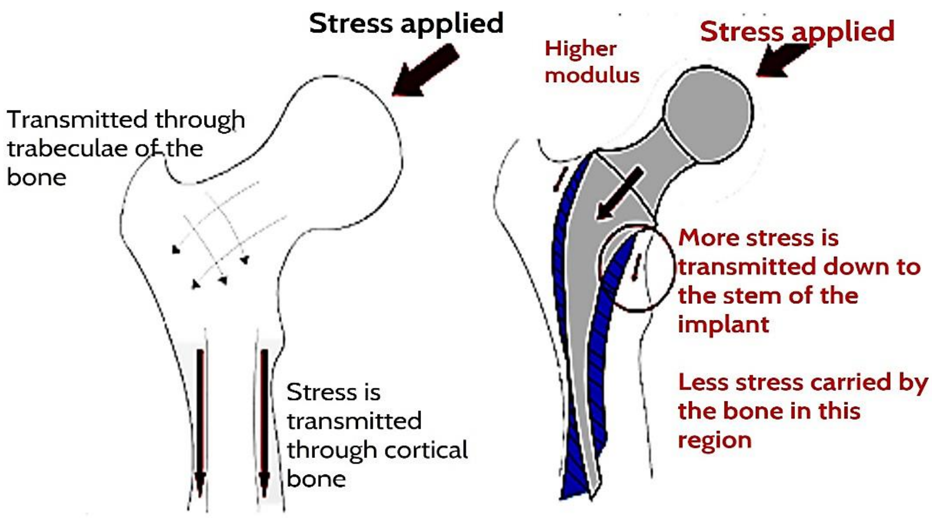

Figure 1.7 Stress shielding phenomenon [66].

Another disadvantage is the mismatch of modulus of elasticity of Ti-6Al-4V alloy (~120 GPa) and the human hard tissue (~30 GPa), that creates stress shielding phenomenon as shown in Figure 1.7 [50]. Due to stress shielding the metallic implant takes the major applied stresses, leaving the more compliant tissue virtually unstressed. Bone resorbs back into the body in this state, and this process is known as disuse atrophy. Furthermore, the stiffness mismatch causes an excessive amount of relative movement between the implant and the bone. This prevents stress from being passed from the implant to the surrounding bone. The contact zone between the implant material and the bone then relaxes, inhibiting new bone production and ingrowth, isolating the implant from its surroundings and limiting osseointegration. Most metallic materials have an elastic modulus that is nearly 10–20 times that of hard tissues.

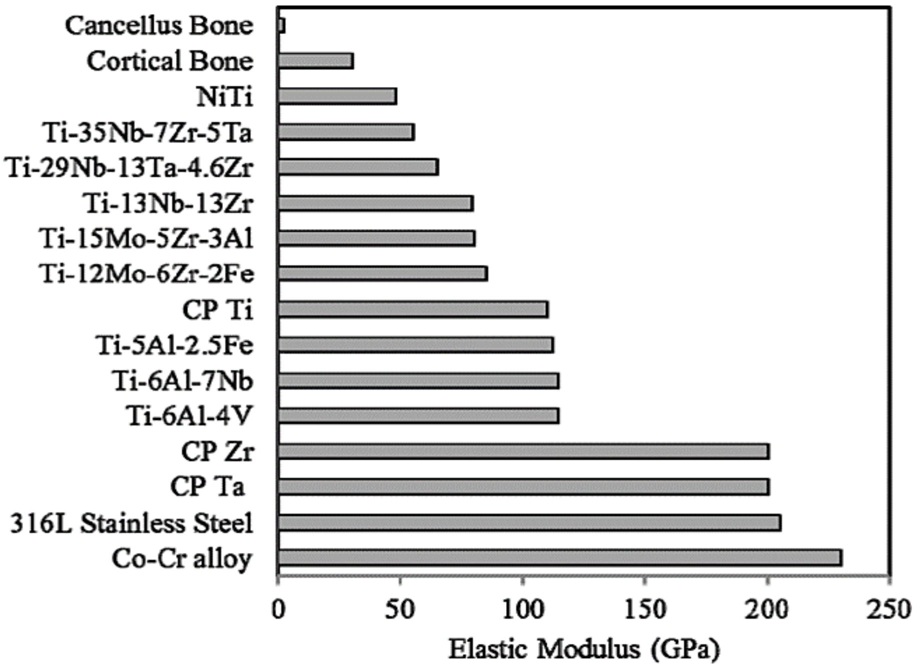

Figure 1.8 Elastic modulus of currently used biomedical alloys [41].

Elastic modulus of some of the commonly used biomedical alloys compared with that of human bone can be observed in Figure 1.8. Most metallic biomaterials are designed for load-bearing orthopaedic joint replacements (hip and knee) and dental implants. Other metallic alloys, such as Co-Cr alloy and 316L stainless steel, have a substantially higher elastic modulus than Ti-based alloys. Elastic modulus of the materials such as stainless steels (190–210 GPa), Co-Cr alloys (210–253 GPa), and Ti-based alloys (55–110 GPa), currently utilised as implant materials is substantially higher than that of the concerned tissues.

Non-toxicity and low elastic modulus of implants are the two crucial requirements for the development of a suitable titanium bio-implant. Much of the biomaterials research has been focused on reducing the elastic modulus of metallic materials for biomedical applications [67,68]. The extremely efficient way of reducing the elastic modulus can be introduction of porosity into titanium and its alloys [69–72]. However, the increase in porosity, leads to loss in strength and would also have deleterious effect on fatigue resistance.

Alloying of titanium with non-allergic/non-toxic beta stabilizing elements such as Nb, Zr, Mo, Ta and Sn shows possibility of controlling the modulus of elasticity through various heat treatments [62,73,74]. Beta titanium alloys, in addition to low elastic modulus also possess optimum mechanical properties and wear resistance for the bio-implants [75,76]. Earlier studies have shown decrease in modulus of elasticity with β and α′ phases formed during sudden quenching from solution treatment temperature [77,78]. With the addition of Nb, V and Ta there is preferential formation of α′′ over α′ on quenching from β phase field. In Ti-Nb type alloy, β phase gets transformed into α′ and α′′ martensites during quenching for the mass% of Nb in the range of ±13% [79]. Recent studies by Liu et al. [80] have also shown low modulus in titanium alloys having fully martensitic structure. Thus, there is possibility of controlling the microstructure by suitable heat treatment in order to achieve lower modulus in titanium alloys. Studies have shown that titanium alloys containing Nb and Zr are more biocompatible than those containing V and Al, and govern the formation of β structure.

1.6 EFFECT OF USSP ON MICROSTRUCTURE

Shot peening is a versatile and effective surface treatment that refines surface grains [81], induces compressive residual stress [82], improves microhardness [83], and enhances corrosion resistance [84]. Many studies have shown that a fine-grained microstructure with a high grain boundary density, promotes passive film formation and improves corrosion resistance. According to Jiang et al. [85], improved corrosion resistance in pure Ti was due to the formation of nanostructure through shot peening.

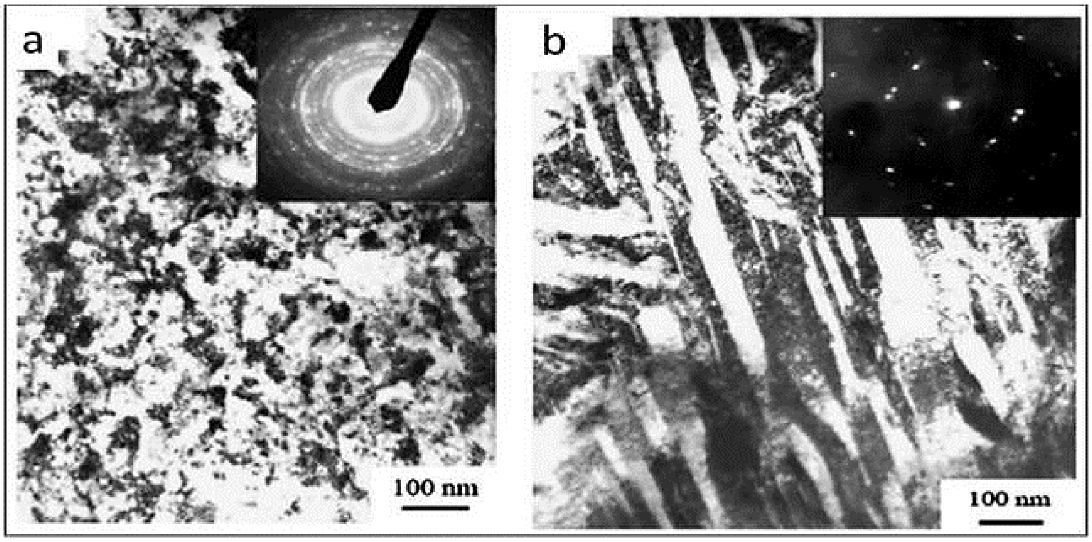

Figure 1.9 Bright field TEM micrographs with related SAED patterns taken at depths of (a) 5 µm and (b) 20 µm from the surface of the alloy Ti–6Al–4V following USSP with 3 mm balls [86].

According to Jelliti et al. [86], the nanocrystalline surface layer of TC4 that was subjected to surface mechanical attrition treatment (SMAT) enhanced the stability of passive film and elevated corrosion potentials. The characteristic ring-shaped SAED patterns of a fine structure may be seen in Figure 1.9a, together with random crystallographic orientations, with an average grain size of 50 nm. Grain size, however, varied from the treated surface towards interior (Figure 1.9b). The process of grain refinement is characterized by a strong twinning activity that subdivides coarse grain into nanograins. The development of nanograins can be linked to high distortions and strain rates during treatment, as well as multidirectional impacts that encourage the activation of several twinning systems. Twinning is thought to have an essential role in grain refining, according to Meng et al. [87]

Nanograins of 10-30 nm were observed by Jin et al. [88] after SMAT treatment of the Ti-Nb-Zr-Fe alloy (Figure 1.10). During the SMAT process, the rapid rise in dislocation density caused by multisystem slip creates high strain energy inside primary grains, which drives localized lattice rotation, resulting in the formation of a significant number of micro and nanocrystals with large angle grain boundaries.

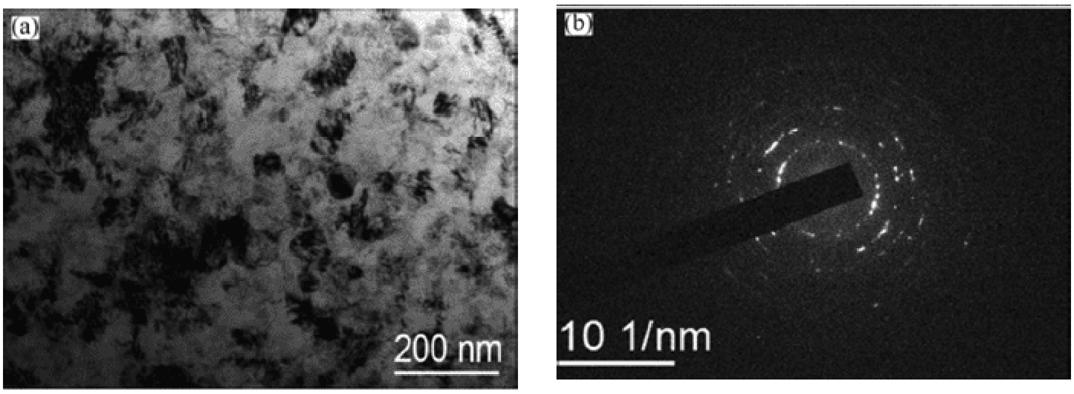

Figure 1.10 (a) TEM micrograph and, the corresponding (b) SAED pattern of Ti-Nb-Zr-Fe alloy after 60 minutes of SMAT treatment by 8 mm steel shots [88].

1.7 CORROSION BEHAVIOUR

There is an increasing interest in specific titanium alloys for application as biomaterial due to their low modulus, excellent biocompatibility, and enhanced corrosion resistance [1,2]. The improved corrosion resistance of such titanium alloys is due to the formation of a stable passive oxide layer at the surface [89]. The electrochemical and physicochemical properties of the passive oxide layer play a vital role in the process of osseointegration and biocompatibility of the titanium implants [90]. The Ti-6Al-4V alloy is commonly used as metallic implants; however, its elastic modulus is high, and the constituent elements V and Al are toxic in nature. Studies have shown that Vanadium ions have high toxicity and are thermodynamically unstable in the human tissue environment [91,92]. This thermodynamic instability is also reflected from the lower corrosion resistance of the Ti-6Al-4V compared to that of Cp-Ti. Aluminium also has been documented toxic in patients undergone hip replacement surgery[93]. Corrosion is one of the main challenges for ensuring the stability and biocompatibility of metallic implants. Therefore, titanium alloys free from Al and V elements have gained more interest in the biomedical field, and more studies have been focused on the β titanium alloys containing non-toxic elements like Nb, Zr, Ta, Sn, and Mo.

Geetha et al. investigated corrosion behaviour of the Ti-13Nb-13Zr alloy which contains non-toxic elements, in various heat-treated conditions and found that the β solution treated and water quenched condition exhibited noble OCP and lower current density among other air-cooled (AC) and furnace-cooled (FC) samples as shown in Figure 1.11a. Formation of stable oxide layer is attributed to the even distribution of alloying elements.

Cvijović et al. [94] compared corrosion behaviour of the Ti-13Nb-13Zr alloy with that of TI-6Al-4V alloy in Ringer's solution and found that both exhibit good passivity but the oxide film formed on Ti-6Al-4V showed better protection against corrosion. Huang et al. [95] compared polarization curves of the Ti-13Nb-13Zr with that of Ti-15Mo (Figure 1.11b) and found that the corrosion potential and corrosion current density of the Ti-13Nb-13Zr was lower than that of the Ti-15Mo and it possessed superior corrosion resistance than that of Ti-15Mo.

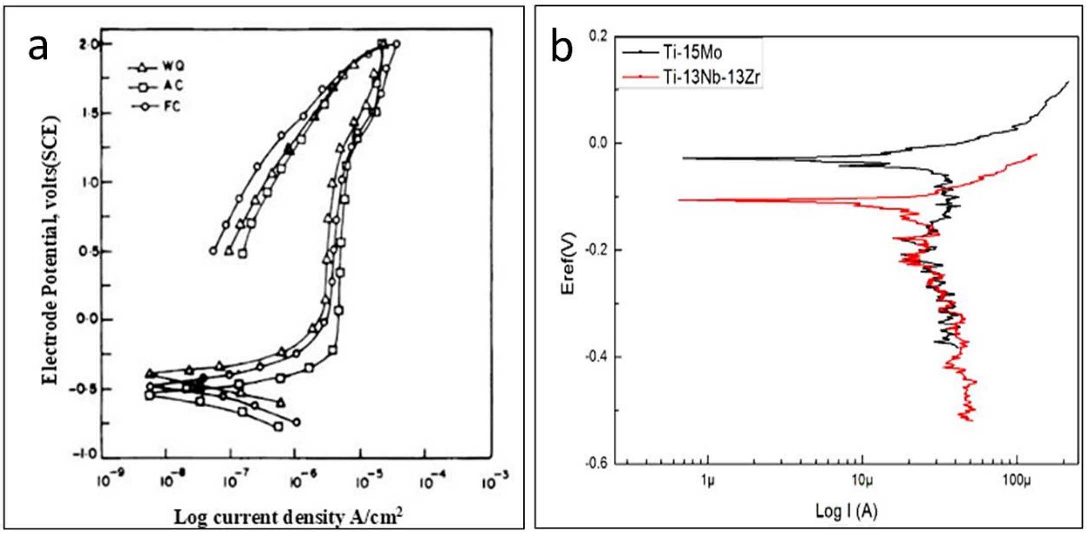

Figure 1.11 (a) Cyclic polarization curves of β solution treated specimens with different cooling conditions of Ti–13Nb–13Zr alloy in Ringer's solution [94]; (b) Polarization curves of Ti-13Nb-13Zr and Ti-15Mo in aerated Ringer's solution [95].

In recent years, Ti-13Nb-13Zr alloy has been studied for modification in microstructure and mechanical properties [96]. Oliveira et al. [97] reported that anodized Ti–13Nb–13Zr alloy does not show any corrosion, even in the chloride-containing solutions. Corrosion of the Ti-13Nb-13Zr alloy results in formation of a passive layer, mainly containing Ti_2O_3, TiO_2, and Nb_2O_5 [98]. According to the study related to electrochemical impedance spectroscopy, titanium alloys develop a double passive layer, namely an outer porous layer, which is important for osseointegration, and a dense inner barrier layer for corrosion resistance [99,100]. The porous layer with high specific surface area possesses the ability of absorption of water and reactive ions from solution [101]. Since the reactive ions can easily penetrate the thin barrier layer, the porous outer layer without any further treatment cannot provide the desired corrosion resistance.

Jindal et al. [102] used ultrasonic shot peening for surface modification to investigate corrosion behaviour of Cp-Ti with surface nanostructure. Corrosion behaviour of nanocrystallized materials has been examined by several researchers. Lei et al. [88] investigated corrosion behaviour on a nanostructured surface of the alloy Ti–Nb–Zr–Fe and discovered that rapid formation of a passive layer increased corrosion resistance on the nanocrystallized surface. According to Garbacz et al. [103], nanostructuring of Cp-Ti reduced corrosion resistance in 0.9 percent NaCl solution as compared to coarse-grained material. Raducanu et al. [104] used rolling to create nanostructure in Ti–Zr–Ta–Nb alloy and found that nanostructuring increased corrosion resistance. Huang et al. [105] investigated the influence of USSP on corrosion rate of a nanostructured Ti–25Nb–3Mo–3Zr–2Sn alloy in NaCl and Ringer's solutions and found that grain refinement lowered the corrosion rate.

Lu et al. [106] proposed several mechanisms for nanocrystallization in different materials and proposed stacking fault energy as one of the important parameters for grain refinement. The grain refinement mechanism in the high stacking fault energy materials (Fe and Al) is mostly by the dislocation-dislocation interactions. In contrast, the low stacking fault energy materials (steel, Cu, Ni-based and Ti-based materials) show twinning as well as dislocation slip as the dominant deformation mechanisms [107]. The key factors that influence the corrosion resistance include grain size, compressive residual stress, surface roughness, and corrosive environment [108–110]. The beneficial effect of compressive residual stress was also shown by Liu et al. [111]. In the samples with compressive residual stresses, the breakdown and re-passivation potential increased with decrease in the current density. Lee et al. [112] showed the effect of surface roughness on corrosion resistance of the 304 SS. With increase in the roughness, the active surface area becomes larger, and the corrosion rate increases. According to Balyanov et al. [113], the ultrafine-grained titanium rapidly forms passive oxide film compared to the coarse-grained titanium.

However, there is limited literature on the effect of surface grain refinement on corrosion behaviour of the biomedical grade titanium. Zhang et al. [114] found that nanograin boundaries and high-density dislocations developed by the USSP treatment helped in creation of channels on the surface for the transfer of ions, thus helps in forming a stable oxide layer. Various techniques have been employed to modify the surface grains of titanium alloys to provide better corrosion resistance and bioactivity [115–117].

1.8 FATIGUE BEHAVIOUR

In fatigue cyclic stress-strain data are known to be dependent on the process of fatigue crack initiation and fatigue crack propagation rate [118]. Since implants are subjected to cyclic stresses throughout their service lives, hence fatigue resistance of the implants material will be critical in predicting the long-term performance of the device [119,120]. Various factors related to surface that affect fatigue life of an implant can be divided into three categories: (i) surface roughness or stress raisers, (ii) changes in the fatigue strength in surface region, and (iii) changes in the residual stress condition of the surface [121–123]. Due to the stress raiser effect and the triaxiality created at the root of the notch, that increase local stress and decrease yielding capacity, of the material the early stage of crack formation gets accelerated.

Lin et al. [124] compared high cycle fatigue behaviour of Ti-7.5Mo with Cp-Ti, Ti-13Nb-13Zr and Ti-6Al-4V alloy in their cast condition. Ti-13Nb-13Zr and Ti–7.5Mo alloys had good strain-controlled fatigue resistance but lower stress-controlled fatigue resistance among the four materials tested, and the fatigue performance was affected by casting induced defects (Figure 1.12). Baptista et al. [125] conducted total strain-controlled fatigue tests on arc melted Ti-13Nb-13Zr alloy at strain amplitudes from 1 to 3.2%. The cold worked and solution treated samples exhibited cyclic hardening.

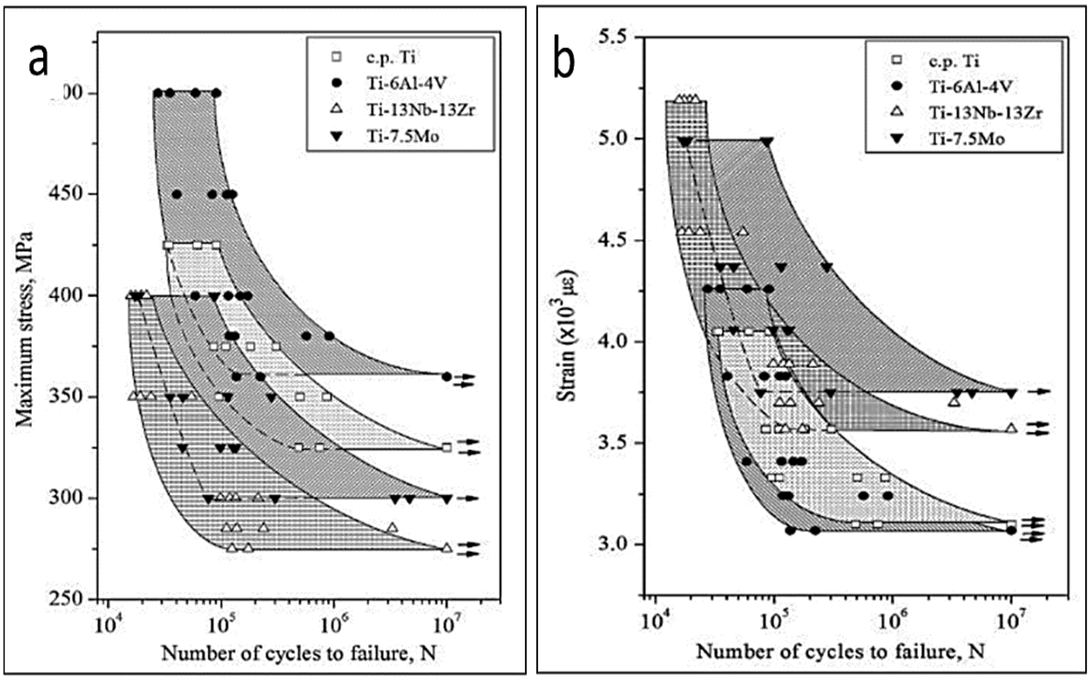

Figure 1.12 (a) Stress controlled fatigue (S-N) curve and, (b) Strain-controlled fatigue data of of Ti-7.5Mo, Ti-13Nb-13Zr, Cp-Ti, and Ti-6Al-4V alloy in their cast condition [124].

The introduction of superficial compressive residual stresses (through blasting, thermomechanical treatments, etc.) in the surface region significantly improves fatigue strength. Due to residual stresses induced during the treatment, surface roughness, defect creation, superficial hardening by plastic deformation, and other factors, the fatigue life of bone engaging implants can be considerably impaired [126]. Fatigue failure mostly originates from the surface or subsurface unless there is bulk defect; therefore, there is much scope for enhancement of fatigue life of structural components through surface modification.

Ultrasonic shot peening (USSP) is one of the highly effective surface modification processes producing surface nanostructure and inducing compressive residual stresses in the surface region, and these changes increase the resistance of the material against crack initiation. In USSP process, the surface of the material is bombarded by spherical steel shots, vibrating at ultrasonic frequency of 20 kHz[107,127]. Due to high frequency of bombardment of shots, the specimen surface gets peened with large number of impacts, in a very short time. USSP causes plastic deformation up to 200-300 µm depth and produces a gradient microstructure, with progressive increase in grain size up to certain depth, affected by the process variables [128]. However, with excessive shot peening, the surface roughness and level of cold working also increase and fatigue performance is reduced. In general, fatigue crack initiation is delayed in fine-grain materials. On the other hand, coarse-grained materials exhibit slower fatigue crack propagation [129,130]. Therefore, it is expected that surface nanostructure would provide better fatigue resistance. The development of nanostructures in the surface region via USSP has been reported in IN718 superalloy[131], titanium TC4 alloy [132], and Ti–6Al–4V alloy [133–135]. Some researchers have found that conventional shot peening improves fretting fatigue life of the Ti–6Al–4V alloy, which they ascribe to induced compressive residual stresses and grain refinement [136,137]. Tsuji et al. [138] investigated the effects of plasma-carburizing and conventional shot peening on high cycle fatigue of Ti–6Al–4V and found that shot peening resulted in a considerable improvement in fatigue life due to associated compressive residual stresses and work hardening in the near surface region. Sanjeev et al. [137] have also shown improvement in low cycle fatigue life of Ti-6Al-4V alloy by ultrasonic shot peening and stress relieving at 400 °C. Shot peening enhanced fretting fatigue life of the Ti–6Al–4V alloy in the high and low cycle fatigue regions under sea water conditions. Fatigue strength of shot-peened Ti–6Al–4V was reduced as the temperature of the stress-relieving treatment was increased from 100 to 260°C [139].

1.9 OSSEOINTEGRATION

The global demand for dental and orthopaedic implants has consistently increased in recent decades, reaching $45.5 billion in 2014 [140–142]. In the 1960s, Branemark [143] investigated osseointegration and implanted the first dental implant. The research and development of dental and orthopaedic implants has continued since then. In the literature, long-term follow-up for various types of implants in patients has been sufficiently recorded [144–146]. Over a 36-year follow-up period, the clinical success rate of dental implants was found to be more than 87.8%, which is mostly due to early bone regeneration [147]. The topography and design of a dental implant are two important aspects that influence its early osseointegration. Dental implants have evolved from hexagonal to conical connections since 1970s, and they are often constructed as rough titanium surfaces [140,148]. Their long-term durability depends on the effectiveness of the connection technique. Conical connections provide superior long-term stability than hexagonal connections. Furthermore, the rough surface improves the surface area of interaction between the implant and the osteoblasts, speeding up bone repair. It also lowers bone resorption by enhancing bonding strength and therefore optimizing interfacial stress distribution, which minimizes dental implant healing time.

During the early phases of osseointegration, two kinds of bone-implant surface interactions are recognized. The first kind includes the creation of a fibrous soft tissue capsule, which results in implant failure if it does not acquire appropriate attachment with the surrounding bone. Osseointegration is the second form, which involves direct bone contact with the implant surface. High fixation is widely acknowledged as one of the requirements for long-term implantation success [149]. The surface qualities have a big impact on the pace of osseointegration and the percentage of bone-to-implant contact [150,151].

Chemical composition, surface energy, roughness, topography, wettability (hydrophilicity/hydrophobicity), and surface shape are all important factors in cell adhesion and survival [152,153]. Biologically and mechanically superior materials, such as commercially pure titanium (Cp-Ti), Ti-6Al-4V, and zirconia, have traditionally been utilized in dental and orthopaedic implants [154,155]. Titanium is one of the most popular implant materials due to its non-toxic nature and biocompatibility. Titanium has a variety of unique features, such as strong fatigue and corrosion resistance in biological fluids [156]. In addition, the β -type alloys have reduced elastic moduli [157,158], better corrosion resistance, and increased biocompatibility [19,159]. The bio-inertness of Ti alloys, on the other hand, causes a longer osseointegration period with bone. Surface treatment technologies can be employed to achieve bioactive surfaces on Ti substrates, overcoming this constraint [160–162]. The shape of implant surfaces at the macro-, micro-, and nano-scales has a significant impact on early bone growth and fixation [163,164].

Several studies have reviewed surface modification strategies for titanium and titanium alloys used in biomedical applications. Mechanical methods such as electropolishing [165], sandblasting, SMAT [166–169]; heat treatment[170]; chemical methods such as acid and alkali etching [171], anodization [172], and sol-gel [173]; chemical vapour deposition methods [174,175], and diamond coatings [176]; and physical deposition methods such as plasma spraying [177], laser deposition [178], plasma treatment [179], sputtering [180], and ion implantation [181]. All of these techniques can be used to alter the chemistry, morphology, and structure of an implant surface. As a result, modified surfaces have potential to increase contact osteogenesis and osseointegration between the implant surface and the host bone.

Furthermore, the most recent literature [182] focuses on macro-, micro-, and nano-scale surface alteration via various approaches with improved osseointegration responses. The multi-scaled morphologies created on the implant promote protein adsorption and osteogenic cell movement, allowing for a faster osseointegration [183]. Liu et al. [184] discovered that nano-materials exhibit distinct surface characteristics due to their increased surface area, increased surface roughness, increased surface flaws and grain boundaries, and changed electron distribution. Nanomaterials are likely to have an impact on the interaction of the surface with proteins due to their unique features, as all proteins are nanoscale entities [102]. Nanoscale surface modification boosts high amounts of IGF-2 (Interleukin Growth Factor-2), BMP-2 (Bone Morphogenic Protein-2), and BMP-6 production by adherent human mesenchymal stem cells over lengthy periods of time in culture. Previous research has found that a nanoscale surface increases cell activity or proliferation. In comparison to traditional implants with smooth surfaces, nanostructured implants have greater attachment and osteoblast function [185,186].

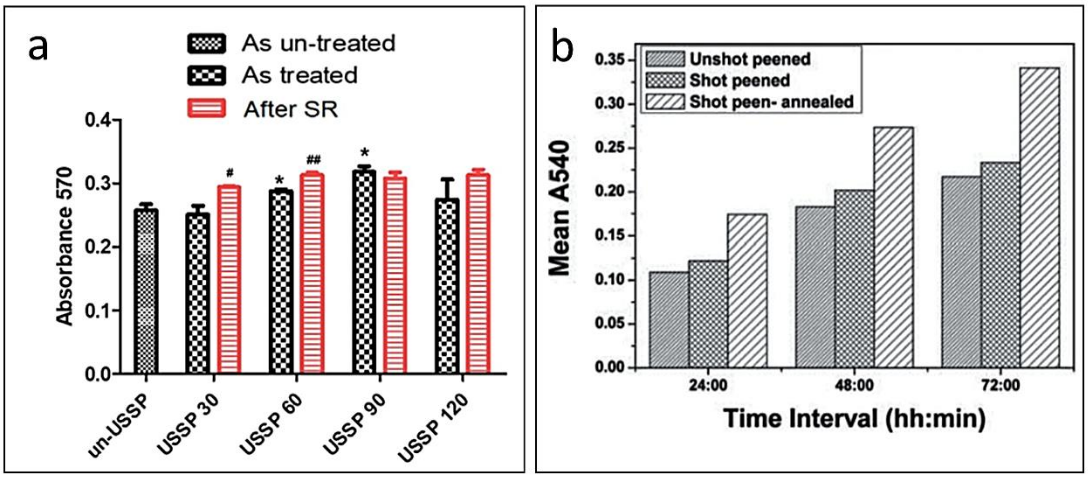

Figure 1.13 Cell cultured on Cp-Ti samples (a) for three days, USSP treated for 30, 60, 90 and 120 seconds; followed by stress relieving [187], (b) for 1, 2 and 3 days, USSP treated for 45 minutes and annealed [102].

Several surface modification approaches have been investigated in recent years to increase implant mechanical characteristics and corrosion resistance. Equal-channel angular pressing (ECAP) [188], ball milling [189], laser shock peening [190], sliding friction treatment [191] and ultrasonic shot peening [192,193], are effective ways of producing ultrafine-grained (UFG) or nano-grained (NG) features in metals. Implant materials with nano-structuring also improve passivation and corrosion resistance. Human Mesenchymal stem cells cultivated by Agrawal et al. [187] and Jindal et al. [102] on Cp-Ti and incubated for different durations, observed substantial increase in cell viability following the USSP treatment (Figure.1.13). Surface nano-structuring, optimal surface roughness, and the presence of a strong positive potential on the surface are thought to be factors in cell development. These macro scale surface modulations have a cumulative influence on cell differentiation, as one would expect. Following the stress-relieving treatment, cell viability was much higher.

1.9.1 Effect of Roughness on Osseointegration

Numerous studies show that the roughness and shape of the implant surface have a significant impact on the osseointegration rate and the quality of the implant bone fixation. The three degrees of surface roughness are macro-scale, micro-scale, and nano-scale. In the macro-scale, topographical roughness varies from millimeters to tens of microns. By roughening the smooth surfaces of implants, the initial attachment and stability can be improved. Furthermore, as compared to smooth surfaces, surfaces with high roughness values result in a greater interlocking reaction in the implant bone contact zone. The roughness value in the micro-scale condition spans between 1 and 10 μm; this roughness range indicates the optimum interlocking reaction between mineralized bone and implants [194].

1.9.2 Effect of Wettability on Osseointegration

Dental implant fixings are often made of Cp-Ti and its alloys. The biocompatibility, corrosion resistance, strength, and osseointegration function of titanium and its alloys make them ideal for use as implant materials. Furthermore, cell activity in the early stages of osseointegration is influenced by the wettability of titanium implant surfaces [153,195–197]. Hydrophilic surfaces (water contact angles ranging from 40° to 70°) are more suited than hydrophobic surfaces on interaction of human body fluids, cells, and tissues with the implant surface [195,198]. The optimal contact angle values and features are still up for debate. Previous studies found that the SMAT process can impact cell-implant interactions by changing the surface properties of implants in terms of grain structure, roughness, topography, chemistry, and wettability [169,199–201].

1.10 SCOPE OF THE PRESENT INVESTIGATION

There is very little information regarding the effect of heat treatments on the elastic modulus and mechanical properties of Ti-13Nb-13Zr alloy. Also, there is no literature regarding systematic study related to impact of USSP on microstructure and corrosion behaviour. In addition, there is no information related to low-cycle fatigue behaviour of surface nanostructured Ti-13Nb-13Zr alloy, especially at smaller strain amplitudes. As a result, a thorough examination of the effect of USSP on microstructural modification, electrochemical corrosion, static immersion corrosion, and LCF of Ti-13Nb-13Zr alloy is warranted. As this alloy is of biomedical importance the effect of USSP on cell proliferation and growth also needs to be examined.

1.11 OBJECTIVES OF THE PRESENT INVESTIGATION

The objectives of the present investigation are listed below:

- Study of the role of thermal treatments on the microstructure, tensile properties and elastic modulus, and establishment of the optimum heat treatment for low elastic modulus of the Ti-13Nb-13Zr alloy.
- Influence of USSP (ultrasonic shot peening) on the microstructure, hardness and residual stress of the material.
- Study of corrosion behaviour and optimization of the USSP parameters for enhancement of corrosion resistance of the alloy in simulated body fluid (Ringer's solution).
- Investigation of the role of ultrasonic shot peening treatment on the low cycle fatigue (LCF) behaviour of the alloy.
- Investigation of biocompatibility of the alloy through cell proliferation/cell viability in Un-USSP, USSP and USSP-SR conditions.