Homi Bhabha National Institute

Ph. D. PROGRAMME

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Synopsis

Superconductors are materials of choice for the production of high field magnets in thermo-nuclear reactors. There are, however, concerns that the Niobium-based materials and Ag sheathed high T_C superconducting materials are not good for such superconducting magnet applications where they may be subjected to long term neutron irradiation. This is because such neutron irradiation would transform them into radioactive materials with very long decay periods [1, 2]. Hence, there is a need for the development of newer superconducting materials with useful properties. In this direction, now the interest has been focused on other transition element alloy superconductors. One such system is the Ti-V alloys which were known to be superconducting for a long time [3]. Previous studies on Ti-V alloys indicate that these alloys are highly machinable and could be used as an alternative material for technological applications [4-6]. However, the usage of Ti-V alloys in the actual technological applications has been rather limited so far, because of the lack of in-depth studies of the superconducting and normal state properties in these materials.

The Ti-V alloys show a variety of structural phases across the complete compositional range [7]. The samples for the present study were chosen in such a way to cover the entire region of the body centered cubic phase of these alloys. It is reported in literature [8] that the superconducting transition temperature T_C increases with increase in the Vanadium concentration among the alloys considered for this study. It is well known that addition of a magnetic impurity in non-transition element based s-wave superconductors suppress the superconductivity due to pair breaking. It is also reported in literature that even the addition of the non magnetic transition elements suppress the T_C [3, 9, 10] due to the formation of localized states. However, it has also been observed that the T_C of a dirty limit superconductor is not affected significantly by disorder [11]. In fact a very high level of disorder is required to change the T_C in such a system [12]. Surprisingly an enhancement in T_C is observed in spite of increased disorder when Ti and V are alloyed, as compared to the T_C of the constituent elements. Such behavior is observed in many transition metal alloys [3]. Apart from the enhanced T_C, these transition metal based disordered binary alloys are also observed to have strong fluctuation conductivity effects well above T_C and well above the upper critical field H_{C2} [13-15]. Moreover, these fluctuations have been found to be independent of the details of sample preparation, surface polishing, the size and shape of samples and the current density [13-15]. This could hint towards the fact that these alloys have the potential of exhibiting even higher values of T_C and H_{C2} than what are observed experimentally at present. However, the reason for the existence of such strong fluctuation conductivity effects well above T_C and well above H_{C2} is not clearly understood.

In the present thesis, the objective is to study in detail the structural, electrical, magnetic and thermal properties of binary Ti-V alloys so as to understand the normal state as well as the superconducting state properties, which might be helpful in resolving the points raised above. We found that the spin fluctuations play an important role in the superconducting and normal state properties [16]. Our studies reveal that the reduction in the electron-phonon interaction as well as the spin fluctuations with the increasing Ti concentration is responsible for the observed variation of the T_C as a function of composition [17]. Apart from this, the structural properties are also observed to influence the normal state and superconducting properties especially the critical current density of these alloys [18, 19]. In addition, several other interesting phenomena such as the high field paramagnetic effect [20], a vortex glass to vortex liquid phase transition [21] and a clear signature of a first order transition in the vortex mater (or the flux line lattice) leading to a peak effect observed in the field dependence of magnetization and the critical current density [22] were also observed in these alloys. The outline of the thesis, which includes the details of these studies are given below.

In **Chapter 1 (Introduction)**, an overview on the current status of the research on Ti-V alloys will be presented. The structural phase diagram of these alloys will be discussed in detail. A brief introduction will be given on the aspects of the superconductivity, which are needed to understand the physical properties addressed in the present work. The motivation of the present work will be given at the end of the chapter.

Chapter 2 (Preparation of samples and experimental techniques) will present the details of sample preparation and experimental techniques used in the present study. The samples of four Ti_xV_{1-x} alloys with x = 0.8, 0.7, 0.6 and 0.4 were prepared by arc-melting the constituent elements in Argon atmosphere. The as-cast ingots were wrapped in Ta-foil and then sealed in quartz ampoules in an atmosphere of Argon. The samples were then annealed at 1300 °C for 10 hours. After that, the samples were cooled slowly to 1000 °C and then quenched rapidly into ice-water from 1000 °C. Details of the structural characterization techniques employed in the present study, such as, X-ray diffraction (XRD) experiments using the synchrotron radiation source, optical metallography, scanning electron microscopy (SEM), and energy dispersive analysis of X-ray (EDAX) will be given in this chapter. Basic principles of the measurements of resistivity, magnetization and heat capacity and the details of the experimental setups used will also be discussed.

A detailed structural characterization of the present alloys has been performed by XRD experiments using the synchrotron radiation source, optical metallography, SEM, and EDAX. The results of such studies will be presented in **Chapter 3 (Structural characterizations)**. The analysis of the XRD patterns performed using the Rietveld refinement technique [16, 18-20] reveals that the major phase in all the alloys is the body centered cubic (bcc) β phase (space group: $Im\bar{3}m$). It is also observed that in Ti rich Ti_xV_{1-x} alloys (x = 0.8 and 0.7) contain secondary phases. The ω phase with a hexagonal crystallographic structure (space group: P6/mmm) is common to both these alloys. However, the α phase with a hexagonal-closed-packed (hcp) crystallographic structure is observed in annealed $Ti_{0.7}V_{0.3}$ alloy (space group: $P6_3/mmc$) whereas stress induced α′ phase with an orthorhombic crystallographic structure is observed in $Ti_{0.8}V_{0.2}$ alloy (space group: Cmcm). The estimated lattice parameters corresponding to these phases are in agreement with the literature [23, 24]. SEM and optical metallography studies on the Ti_xV_{1-x} alloys reveal that the major β phase of these alloys consists of well connected grains of varying grain size. The average size of the grains in these alloys ranges from few tens to few hundreds of μm. The signature of the α phase in the annealed $Ti_{0.7}V_{0.3}$ alloy and the stress induced α′ phase in both the as-cast and annealed samples of the $Ti_{0.8}V_{0.2}$ alloy is also visible in both optical and electron micrographs. In all the Ti_xV_{1-x} alloys, etch pits are also visible and these etched pits are distributed uniformly all over the sample. The results of the EDAX experiments show that the fluctuation in compositions of the investigated alloys over the entire sample surface is less than 2 % of the nominal compositions of the alloys.

In **Chapter 4 (Superconducting properties of Ti_xV_{1-x} alloys),** the details of the superconducting properties of the Ti_xV_{1-x} alloys will be presented. The superconducting transition temperature T_C is estimated from the temperature dependence of resistivity, magnetization and heat capacity measurements. The estimated T_C values increase from 4.12 to 7.34 K as x is decreased from 0.8 to 0.4. These values are in agreement with the previously published results [8, 25]. The upper critical field H_{C2} and the lower critical field H_{C1} at different constant temperatures are determined from the isothermal M versus H curves obtained at various constant temperatures below the T_C of these alloys. The highest H_{C2}

value is obtained in the $Ti_{0.6}V_{0.4}$ alloy (H_{C2} is about 10 T at T = 4.2 K). The density of states at Fermi level $N(0)^{HC2}$ is estimated from the slope of the experimental $H_{C2}(T)$ curve near the T_C [26]. Electronic band structure calculations have been performed to estimate the density of states at the Fermi level $N(0)^{BS}$. The $N(0)^{HC2}$ is found to be considerably larger than the $N(0)^{BS}$ for all the Ti_xV_{1-x} alloys. It is well known that the electron-phonon interaction leads to renormalization of the density of states at the Fermi level. The renormalization factor is $(1 + \lambda_{ep})$, where λ_{ep} is the electron-phonon coupling constant. The values of $N(0)^{HC2}$ and $N(0)^{BS}$ are used to estimate λ_{ep} from the relation: $N(0)^{HC2} = N(0)^{BS} (1+\lambda_{ep})$ [26]. The estimated value of λ_{ep} increases from ~0.5 to ~1.0 as x is decreased from 0.8 to 0.4. The Maki parameter α_M estimated for these Ti_xV_{1-x} alloys is higher than unity, implying that the Pauli paramagnetic pair breaking effect significantly influences the upper critical field in these alloys. Strong electron-phonon interactions, however, reduce the relative importance of Pauli paramagnetic pair breaking effect in V rich alloys. The experimental $H_{C2}(T)$ data are then analyzed with the formalism given by Orlando et al. [27], which considers both the Pauli paramagnetic pair breaking effect and the corrections for the electron-phonon interactions. The magnitude and the temperature dependence of H_{C1} are found to be nearly consistent with the predictions of the Ginzburg-Landau-Abrikosov-Gor'kov (GLAG) theory [28-31]. Two fundamental superconducting length scales namely the Ginzburg-Landau coherence length $\xi_{GL}(0)$ and the Ginzburg-Landau London penetration depth $\lambda_{GL}(0)$ at absolute zero temperature are estimated using the Ginzburg-Landau relations. The values of $\xi_{GL}(0)$ for these the Ti_xV_{1-x} alloys come out to be in the range of ~48-60 Å, which are considerably larger than the estimated mean free path for the electron conduction (l_e) in these alloys. This indicates that the Ti_xV_{1-x} alloys are dirty limit superconductors. It is found that the Ti_xV_{1-x} alloys are characterized with very high values of Ginzburg-Landau parameter $\kappa = \lambda_{GL}(0) / \xi_{GL}(0)$ and the κ value increases with the increase in x. For example, the value of κ is as high as ~60 for the $Ti_{0.7}V_{0.3}$ alloy. Hence, these alloys are extreme type-II superconductors. The thermodynamic critical field H_C is deduced from the measured electronic heat capacity in the superconducting state [32]. The experimental $H_C(T)$ curves are fitted with the empirical relation $H_C(T) = H_C(0) [1-(T/T_C)^2]$ to obtain the value of $H_C(0)$. The obtained value of $H_C(0)$ in the Ti_xV_{1-x} alloys increases with decreasing x. Furthermore, we have estimated the Ginzburg number G_i [33] for the present Ti_xV_{1-x} alloys. The G_i number increases with increasing x and their values are in the range $\sim 10^{-6} - 10^{-5}$ [19]. Although these values are lower than those for the high-T_C Cuprate superconductors ($G_i \sim 10^{-2}$), they are considerably higher than those for typical low-T_C superconductors ($G_i \sim 10^{-8} - 10^{-9}$). Thus, significant thermal fluctuation effect is expected in these crystalline Ti_xV_{1-x} alloys in spite of their low values of T_C.

The studies on the effect of superconducting fluctuations in the Ti_xV_{1-x} alloys above the T_C will be presented in **Chapter 5** (**Fluctuation conductivity in Ti_xV_{1-x} alloys**). The strong rounding-off behavior of $\rho(T)$ curve observed above T_C as well as the relatively high positive magneto-resistance observed in the temperature regime between T_C and ~2T_C for the present Ti_xV_{1-x} alloys are due to the superconducting fluctuations. Experimentally obtained excess conductivity is then analyzed with the help of the existing theoretical models of Aslamazov-Larkin [34] and Maki-Thompson [35-37]. For all the Ti_xV_{1-x} alloys except $Ti_{0.8}V_{0.2}$, the magnitude and the temperature dependence of the excess conductivity at low reduced temperatures ($\varepsilon \leq 0.1$) are found to be well explained by the Aslamazov-Larkin theory for 3D superconducting fluctuations. The roles of different pair-breaking mechanisms, such as, thermal phonons, magnetic impurities and spin fluctuations in the complete suppression of the Maki-Thompson type contribution to superconducting fluctuations in Ti_xV_{1-x} alloys will be discussed. It is observed that thermal phonons may not be strong enough for the complete suppression of the Maki-Thompson type superconducting fluctuations in these alloys. A linear field dependence of magnetization is observed in the normal state of these alloys up to magnetic fields of 80 kOe, which suggests that these alloys may not contain any magnetic impurities. Hence, the only probable pair breaking mechanism that leads to the suppression of Maki-Thompson type superconducting fluctuations in Ti_xV_{1-x} alloys is the spin fluctuations. Therefore, motivated by the fact that the spin fluctuations might be important in the Ti_xV_{1-x} alloys we have performed a detailed study of the normal state properties of these alloys.

Chapter 6 (Normal state properties of Ti_xV_{1-x} alloys) will present the study on thermal, electric transport and magnetic properties of the Ti_xV_{1-x} alloys in their normal state. The heat capacity measured at low temperatures above T_C is fitted with the function $C(T) = \gamma T + \beta T^3$ to obtain the Sommerfeld coefficient of electronic heat capacity γ and the Debye temperature θ_D. The electron-phonon coupling constant λ_{ep} is also estimated using the experimental γ value and the density of states at Fermi level $N(0)^{BS}$ determined from the band structure calculations. The electrical resistivity in the Ti_xV_{1-x} alloys with x = 0.7 and 0.8 increases with decreasing temperature over a wide range of temperatures. For the Ti_xV_{1-x} alloys with x = 0.4 and 0.6, the electrical resistivity in the normal state increases with increasing temperature at all measured temperatures up to room temperature. At low temperatures ($15 < T < 40$ K), the temperature dependence of electrical resistivity observed for these two alloys is found to be described well with the function: $\rho(T) = \rho_0 + AT^2 + BT^5$. The T^5 term represents the phononic contribution to the resistivity at low temperatures. The coefficient of the T^5 term is found to be unusually small and it is positive for the x = 0.4 alloy and negative for the x = 0.6 alloy. This behavior and the quadratic temperature dependence of low-temperature resistivity are characteristic feature of the spin fluctuations [38]. In the normal state, the Ti_xV_{1-x} alloys exhibit temperature induced dc magnetic susceptibility $\chi(T) \propto -T^2 \ln T$, which also indicates the presence of the spin fluctuations [39, 40]. We have also estimated the Stoner factor S for the Ti_xV_{1-x} alloys. The S is observed to be about ~2 for the x

= 0.6 and 0.4 alloys. Such high value of S is generally observed in materials with spin fluctuations [41, 42]. Kadowaki-Woods scaling relation [43] between γ^2 and the coefficient A of the quadratic term of the low-temperature electrical resistivity is also observed to be valid for the Ti_xV_{1-x} alloys (for x = 0.6 and 0.4). The above experimental evidences clearly suggest the presence of spin fluctuations in the Ti_xV_{1-x} alloys. Our study also reveals that the spin fluctuations present in these alloys are itinerant in nature.

We have further studied the influence of spin fluctuations on the superconductivity in Ti_xV_{1-x} alloys. When spin fluctuation interactions are not important, the superconducting transition temperature T_C of a superconductor is governed by three important parameters, namely, θ_D, λ_{ep} and the coulomb interaction parameter μ^* [44]. The calculated T_{C0} values using the McMillan formula for V rich Ti_xV_{1-x} alloys are found to be significantly higher than the experimentally observed values (T_C). The disagreement between T_{C0} and T_C increases with decreasing x. This observed disagreement arises mainly due to the electron-spin fluctuation interactions [45-48]. In the other words, we can say that the spin fluctuations in Ti_xV_{1-x} substantially reduce the superconducting transition temperature from the theoretically predicted value (T_{C0}) to the one observed experimentally (T_C). We have also provided an explanation based on the distribution of the electron-spin fluctuation interaction for the observed fluctuation conductivity above T_C.

The commercial application of a superconductor depends on its capability of carrying dissipation-less current in the presence of high magnetic fields. This aspect will be discussed for the Ti_xV_{1-x} alloys in **Chapter 7** (**Critical current and flux-line pinning in Ti_xV_{1-x} alloys**). This chapter will be divided in two parts. The first part will cover a detailed study on the field dependence the critical current density J_C and the pinning force density F_P in both the as-cast and annealed samples of the Ti_xV_{1-x} alloys. The peak effect (PE) observed in the field dependence of magnetization curves [M(H) curves] in high fields near H_{C2} will be discussed in the second part of chapter 7. Isothermal M(H) curves obtained for both the as-cast and annealed samples of the Ti_xV_{1-x} alloys at various constant temperatures below their respective T_C's are distinctly irreversible. The observed irreversibility is caused by the pinning of flux lines within the superconductors. We have estimated the J_C from the irreversible M(H) curves with the help of the Bean's critical state model [49]. The as-cast $Ti_{0.7}V_{0.3}$ alloy has the highest J_C value among all the investigated alloys. In zero fields and at 2 K temperature, the J_C value estimated for this alloy is ~6 × 10^8 A/m^2. In order to understand the pinning mechanisms operating in these superconducting Ti_xV_{1-x} alloys, a detailed analysis of the field dependence of pinning force density $F_P = J_C \times H$ is done using the Dew-Hughes model [50]. Except in the as-cast and annealed samples of the $Ti_{0.8}V_{0.2}$ alloy, the pinning force in all the Ti_xV_{1-x} alloys (both the as-cast and annealed) in the field regime of the main magnetic irreversibility arises primarily from the flux-line pinning by normal surface pins [19, 20]. The grain boundaries, edge dislocations and martensitic α phase boundaries (in annealed $Ti_{0.7}V_{0.3}$ alloy) constitute the sources of such normal surface pinning centers in these alloys [19, 20]. In the as-cast and annealed samples of the $Ti_{0.8}V_{0.2}$ alloy, flux-line pinning occurs predominantly at normal point pins while the role of normal surface pins is prominent at relatively lower fields [19]. Substantial amount of ω phase available in the as-cast and annealed samples of these alloys functions as normal point pinning centers [19]. We will then present the effect of thermal fluctuations on the high field critical current density in these alloys. The peak effect is observed in the isothermal M(H) curves in high-field regime near H_{C2} in all the present alloys except annealed $Ti_{0.7}V_{0.3}$. The peak effect in various superconductors occurs due to a field induced transition in the flux-line lattice from a phase of low to high flux pinning characteristic [51]. The nature of this phase transition has been investigated by using a minor hysteresis loop (MHL) technique [51**]** which revealed various characteristic features i. e., matastability, and superheating/supercooling associated with a first order phase transition. Moreover, we have estimated the equilibrium magnetization (M_{eq}), which exhibits a clear jump in the PE regime. This was used for the estimation of the latent heat with the help of the Clausius-Clapeyron relation. At 2 K, latent heat comes out to be L ~35.7 μJ/g for the annealed $Ti_{0.8}V_{0.2}$ alloy whereas L~70 μJ/g at 4 K for annealed $Ti_{0.4}V_{0.6}$ alloy. These results provide further support that the PE in the Ti_xV_{1-x} alloys is associated with a first order phase transition in the vortex matter.

In **Chapter 8 (Vortex-glass to vortex-liquid transition in annealed $Ti_{0.7}V_{0.3}$ alloy),** we will present the experimental study of vortex-solid to vortex-liquid phase transition [52-54] in the $Ti_{0.7}V_{0.3}$ alloy through the measurement of electrical resistivity in presence of various constant magnetic fields up to 50 kOe. We found that both the width of the normal to superconducting phase transition in the alloy, and the tail in the electrical resistivity observed before achieving the zero resistivity state increase with increasing magnetic field. Using the Arrhenius relation, we have identified a vortex-glass to vortex-liquid phase transition in this low T_C superconductor. We have also identified a critical region corresponding to this phase transition and obtained the critical exponent for the same. We have used a modified vortex-glass model to separate out the temperature and field dependent parts of the effective pinning energy. The field dependent part of the effective pinning energy exhibits power law dependence on the applied magnetic field, and both the temperature and field dependent parts of the effective pinning energy indicate a crossover of behavior close to the vortex-glass to vortex-liquid phase transition in the alloy. This change of behavior might be because of a crossover from a regime of single vortex pinning dominate to a regime of collective vortex pinning tends to dominate.

In **Chapter 9 (High-field paramagnetic Meissner effect in Ti_xV_{1-x} alloys),** we will present the study related to an anomalous feature in the samples of annealed $Ti_{0.7}V_{0.3}$ and as-cast as well as annealed $Ti_{0.8}V_{0.2}$ alloys. In contrast to conventional type-II superconductor, the magnetization in these samples is observed to increase when temperature is decreased below the T_C in presence of high magnetic fields beyond a certain critical value. In this field and temperature regime, it is observed that the magnetization values in the field cooled cooling (FCC) protocol have a smaller magnitude as compared to that in the field cooled warming (FCW) protocol. We have also observed that the magnetization at a constant temperature well below T_C when cooled in the presence of high magnetic fields increases with time. These observed features are characteristics of the high-field paramagnetic Meissner effect (HFPME) [55-58]. We argue that the HFPME is observed in these alloys due to the non-uniform flux-line pinning at the boundary of the inhomogeneously distributed α' or α phases [20]. As stated in Chapter 2, the α' phase in both the as-cast and annealed samples of $Ti_{0.8}V_{0.2}$ alloy is a stress induced phase which forms during the mechanical processing such as cutting of the sample. We therefore remove the α' phase from the annealed sample of $Ti_{0.8}V_{0.2}$ alloy, which shows the signature of HFPME, by carrying out a second stage annealing following the same protocol employed during the first annealing [20]. HFPME becomes completely suppressed in this re-annealed sample suggesting that the non-uniform flux density promoted by pinning at α' or α phase is indeed the reason for the occurrence of HFPME in $Ti_{0.8}V_{0.2}$ and $Ti_{0.7}V_{0.3}$ alloys. Our studies suggest that the observation of HFPME in these alloys is due to the inhomogeneous flux pinning and the trapping of the flux lines at the α' or α phase boundary, which creep from rest of the sample volume.

In **Chapter 10 (Summary, Conclusion and Future work),** we will present the summary and the conclusions drawn from the study and the scope for the further studies. The important conclusions are listed below:

(1) The superconducting transition temperature T_C of the present alloys is higher than the constituent elements Ti and V. The experimentally observed $T_{C,}$ however, is much less than that estimated by considering the electron-phonon interaction alone.

(2) The Ginzburg number G_i for these Ti_xV_{1-x} alloys are estimated to be about $10^{-6} – 10^{-5}$ indicating the significant influence of the thermal fluctuations in these alloys. Such thermal fluctuations contribute to the observation of fluctuation conductivity well above T_C.

(3) The presence of spin fluctuations in V rich Ti_xV_{1-x} alloys is inferred from the normal state properties. We have shown that the spin fluctuations present in these alloys are itinerant in nature.

(4) The variation of T_C with composition in the Ti_xV_{1-x} alloys is explained by considering the electron-phonon interaction and spin fluctuations. We also provide an explanation based on the distribution of the electron-spin fluctuation interaction for the observed fluctuation conductivity above T_C.

(5) Grain boundaries, edge dislocations, ω phase, and α (or α') phase boundaries seem to be the sources of the flux-line pinning mechanisms in these superconducting alloys. The irreversibility field H_{irr} particularly in Ti rich Ti_xV_{1-x} alloys is observed to be lower than H_{C2} due to the increased role of thermal fluctuations which ultimately resulted in the suppression of the high-field J_C in these alloys.

(6) The relatively strong flux line pinning along with thermal fluctuation effects in annealed $Ti_{0.7}V_{0.3}$ alloy resulted in the formation of the Bose Glass phase in the mixed state of this superconductor. A difference is observed between the observed properties at high and low field regime when the data is analyzed with the existing scaling law. A new scaling law is proposed to resolve this difference.

(7) We have observed the high field paramagnetic effect in $Ti_{0.8}V_{0.2}$ alloy and annealed $Ti_{0.7}V_{0.3}$ alloy. We have shown that this effect results from the inhomogeneous flux pinning due to the presence of stress induced martensitic α' phase in $Ti_{0.8}V_{0.2}$ alloy, whereas it is resulted from the inhomogeneous flux pinning due to the presence of α phase in $Ti_{0.7}V_{0.3}$ alloy. We have shown that the high field paramagnetic effect is related with the flux pinning and can result wherever the inhomogeneous pinning centers are present.

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