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IDENTIFICATION OF DISLOCATIONS, GRAIN BOUNDARY SLIDING AND POINT DEFECTS WITH THE AID OF SEM ANALYSIS

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Abstract— There has been a consistent look for new materials for high temperature applications, for example, for parts of steam generators, parts of warm power plants, funneling of fluid metal reactors, which are probably going to be presented to temperature 550 ^{0}C or more. Great quality and pliability values controlled by uncommon steels settle on them the primary selection of fashioners for applications specified. Changed 9Cr- 1Mo steel has been being used for such applications since 10 years. For any given material, alongside the mechanical properties, there is a need to decide the crawl property if the material is the decision for high temperature benefit applications. Encourage, as it has been encountered that the welds are likely zones of disappointment, under outrageous states of load and temperature, testing the crawl property of the welded joints of a given material turns into an essential part of research. Consequently it is proposed to test into the crawl resistance part of the welded joints of 9Cr-1Mo steel under various states of load and temperature.

Index Terms— Crawl property (Creep), 9Cr-1Mo Steel, SEM.

I. INTRODUCTION

There has been a consistent scan for new materials for high temperature applications, for example, for parts of steam generators, parts of warm power plants, funneling of fluid metal reactors, which are probably going to be presented to temperature 550^{0}C or more. Great quality and flexibility values controlled by unique steels settle on them the principal selection of creators for applications specified. Altered 9Cr-1Mo steel has been being used for such applications since 10 years. For any given material, alongside the mechanical properties, there is a need to decide the crawl property if the material is the decision for high temperature benefit applications. Assist, as it has been encountered that the welds are likely zones of disappointment, under extraordinary states of load and temperature, testing the crawl property of the welded joints of a given material turns into a vital part of research. Therefore it is important to concentrate the crawl resistance of the welded joints of 9Cr 1Mo steel with various burdens and temperature. While malleable crawl tests are tedious and bulky, the space crawl tests can help the specialist to concentrate the crawl property of a given material in a shorter time term. Space or Impression crawl tests are led utilizing hard indenter utilized as punch with tests directed at various burdens. Under each test, strain experienced by the specimen is plotted against time, to get the crawl bend which is known to have balanced correspondence with its ductile test crawl bend. Temperature and burdens for the test may fluctuate to reproduce down to earth conditions. Different specialists have taken endeavors to associate basic elements like disengagement thickness, grain size and nature of grain limits to the miss happening design under the punch. The favorable position this approach is that a little example is sufficient to survey the crawl properties, in this way enabling the examiner to concentrate the neighborhood variety in properties of a bigger specimen. It was proposed to age the specimens of 9Cr-1Mo steel under various conditions so that variety of sneak properties in both the parent and the weld metal could be examined embracing the space crawl test system. Tests are intended to concentrate some data with respect to basic crumbling of the material. Furthermore, filtering electron microscopy strategy was received to watch the small scale basic changes and relationship technique was endeavored to connection them to the watched crawl conduct. Space crawl studies are embraced for different metal and composite classes in various research facilities of the nation and information accessible on exceptional application steels are restricted. Considering the significance of 9 Cr-1Mo steel, the present review was embraced.

II. LITERATURE REVIEW

Numerous unique steels are considered for making high temperature auxiliary segments –say warm exchanger tubes , primary/warm steam channels, header and warmth recuperation funnels –to name a couple. In greater part of the instances of the use of the segments said , change in vitality effectiveness is achieved by expanding the steam temperature which thus requests high temperature opposing materials (which infers crawl opposing materials) to be placed being used. 9 Cr-1 Mo is one such steel which can be utilized for the said applications. Whenever Niobium (Nb) and Vanadium (V) is included ideal extents, the steel is alluded to as adjusted 9 Cr-1 Mo steel. The steel is under ASTM/ASME Grade 91 and its crawl quality should be explored in detail and a portion of the accessible papers are introduced here with the discoveries recorded.

1. K.Kimura, K.Sawada,H.Kushima, Y.Toda-Long term crawl quality of ASTM/ASME review 91 steels was examined by them. Nearness of delta ferrite stage was watched. The higher nickel containing heat shows bring down sneak burst quality in the long haul benefit at 6000C, in spite of the fact that nickel focus was not as much as the most extreme indicated in the review. Homogeneously recouped sub-grain structure was seen on the examples crawl burst after around 80,000 hours at 6000C, for both high nickel low quality warmth and low nickel high quality one. Just few fine MX carbo-nitride particles with an expansive number of coarse Z-stage were seen on the crawl burst example of high nickel low quality warmth, as opposed to low nickel high quality warmth in which numerous MX particles were as yet watched and Z stage arrangement was not articulated. The distinction in solidness of fine MX carbo-nitride particles amid crawl presentation at the hoisted temperatures is a reason for warmth to-warmth variety of long haul crawl quality of these steels, watch the specialists. Diminish in stage change temperature of Ac1 with increment in nickel substance may decrease steadiness of the accelerates at the hoisted temperatures. Nickel substance ought to be decreased with a specific end goal to supress an extensive drop in long haul crawl quality of Grade 91 steel.

2. Yuta Tanaka, Keiji Kubushiro, Satoshi Takahashi, Noriko Saito, Hirokatsu Nakagawa –studied the development of Z-stage in 9-12% Cr modern steels and watched that this Z-stage is principally a Cr-V based nitride, additionally containing little amounts of Nb and Fe. They have concentrated the microstructural development (carbide coarsening , Laves stage nucleation and development) of these steels over long haul exposures. Assist, they have set up the erosion oxidation conduct of the steels. The unpredicted untimely disappointment of steels after a long haul presentation is because of the presence of Z-stage, according to the conclusions drawn by these specialists.

3. Yuta Tanaka, Keiji Kubushiro, Satoshi Takahashi, Noriko Saito, Hirokatsu Nakagawa-The crawl harm procedure of high Cr steel welded joints is portrayed by the arrangement and development of crawl voids preceding the start of breaking and this development and development represents a huge extent of crawl life, as indicated by the examiners. A nitty gritty examination concerning the discovery and quantitative assessment of crawl voids and smaller scale breaks in welded joints for use in outstanding life appraisal is made. A crawl test on an extensive welded joint in changed 9Cr-1 Mo steel was led under 60 MPa at 650 0C. The perception of the microstructure in the warmth influenced zone (HAZ) was made for the example hindered in the crawl tests at 25% of a break life. The microstructure around the crawl void was described by the specialists utilizing an electron backscatter diffraction design (EBSD) technique. It was likewise seen by them that the crawl voids framed and created along arbitrary high point grain limits. At first framed void advanced particular dynamic recuperation and dynamic recrystallization in its encompassing microstructure, trailed by sub-limit development.

4. S. Latha, M.D. Mathew, P. Parameswaran, K.Laha, S. PannerSelvi, S.L.Mannan – Creep properties of 14Cr-15 Ni-Ti changed steel, alloyed with phosphorus and silicon were researched at 9730K in the anxiety run 175-250 MPa. The phosphorus content in the combinations was 0.025 and 0.04 wt%, silicon 0.75 and 0.95 wt% and titanium in the range 0.16-0.3 wt %. The variety between least crawl rate and burst life for these combinations was found to take after the Modified Monkman Grant relationship. The opposite of Modified Monkman Grant relationship which is characterized as the harm resistance parameter was observed to be over ten for these compounds, demonstrating that these combinations can withstand high strain fixations. Optical infinitesimal examination uncovered broad network misshapening and precipitation. Sneak harm as breaks or holes was not seen in these combinations validating high resilience for crawl harm. Expansion of boron was found to avert grain limit harm.

5. D.P. RaoPalaparti, E. Isaac Samuel, B.K.Choudhary, M.D. Mathew-Creep crack properties of T91 steam generator tube steel have been inspected by the specialists at 9230K in standardized and tempered condition in the anxiety scope of 55-150 MPa. At all anxiety conditions, the crawl disfigurement was portrayed by an abatement in sneak rate in the transient crawl taken after by a base sneak rate in the auxiliary crawl arrange and a fast increment in sneak rate in tertiary crawl organize. A deliberate decline in crawl rate with diminishing anxiety was seen in transient, optional and tertiary crawl. Stretch reliance of least crawl rate and crack life obeyed control law crawl. Both least crawl rate and crack life showed deviations as far as lower individual anxiety type values at low worries than those gotten at high burdens. A diminishing in crawl malleability was seen with increment in crack life. The crack mode remained trans-granular at all test conditions. Crawl break quality of SG tube steel has been observed to be tantamount to those detailed in writing and additionally to those said in the standard plan codes.

6. Andreas Klenk, Magdalena Speicher, Karl Maile – concentrated the weld conduct of martensitic steels and Ni-based amalgams for high temperature parts utilized as a part of the profoundly effective steam control plants where working temperature is constrained to 7000C and weight 350 bar. The examinations included investigation of the sneak conduct before disappointment in regard of the welded area/parent metal locale of ferritic/martensitic steels T24, T/P92 and VM12 to settle these steels' reasonableness for the given application. Weld quality components are talked about and data on precipitation and disengagement state in the virgin and matured conditions are exhibited by the agents.

7. FujimitsuMasuyama – has displayed some of his discoveries on microstructural crawl debasement, the hardness crawl life demonstrate and the rearranged tertiary crawl displaying of the Omega technique for crawl life evaluation of crawl quality upgraded ferritic (CSEF) steels, for example, Gr. 91,92 and Gr.122. In these steels, martensitic structure made out of fine martensite strip, square, parcel and earlier austenite grains ; precipitation and disengagement structures.

III. DISLOCATIONS AND MECHANISMS OF CREEP IN SINGLE PHASE MATERIALS

The first mechanism of creep in single phase materials is slip followed by climb. When a stress above a critical value is applied to a metal crystal, the crystal lattice slips by lines which are called as dislocations. A dislocation occurs first in any of the closed packed planes since this requires the least energy. If the edge of the plane is the line of dislocation, it is known as ‘’edge dislocation’’ consisting of unfinished atomic plane. If the Burger vector is parallel to the dislocation line and can slip on any close packed plane in the crystal, the dislocation is called as screw dislocation. Figure 1.1(a) shows the slip of the edge dislocation slipping through a crystal lattice and producing a unit of slip called slip step, on the surface of the crystal and figure 1.1(b), a typical screw dislocation [1].

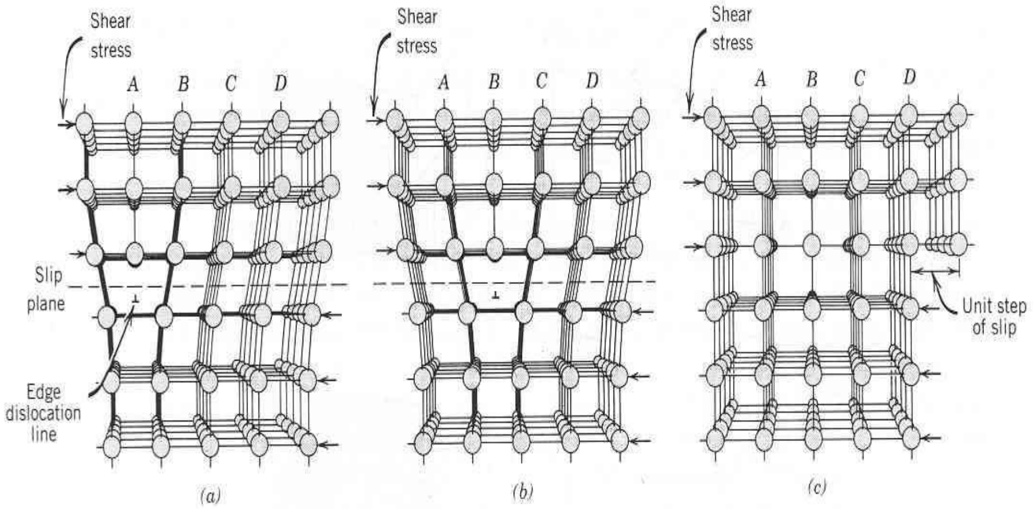

Fig. 1. 1 (a) Edge dislocation

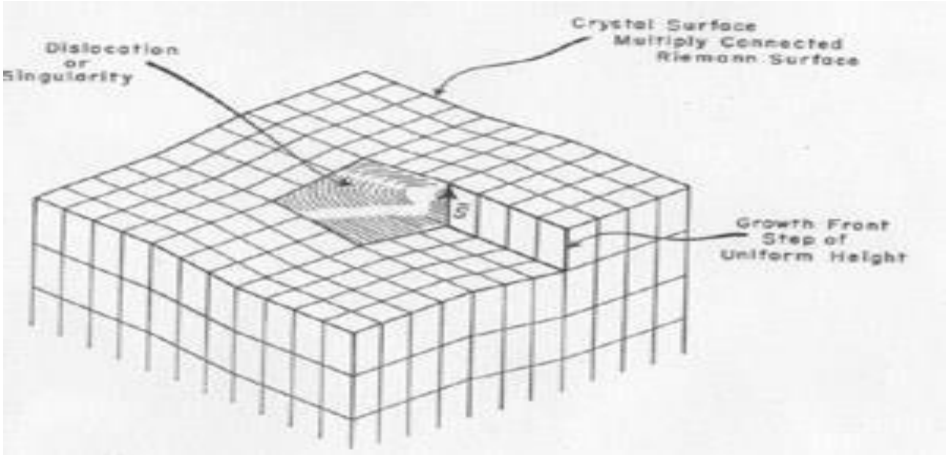

Fig. 1.1(b) Screw dislocations

Slip will occur when the shear stress exceeds the critical resolved shear stress as the stress is applied in a given polycrystalline metal resulting in deformation. Jogs are the leaving slip steps when dislocations cut each other. If the dislocations are immobile the jogs can hinder slip [2].

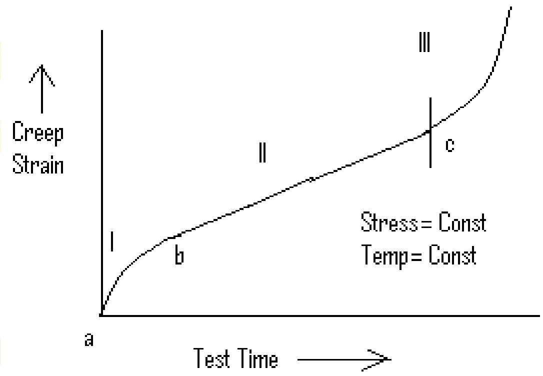

Fig 2.1 Typical creep Curve for Materials- Stages I: Primary Creep; II: SecondaryCreep; III: Tertiary Creep

Figure 2.1 depicts typical creep curve for materials. In the primary creep stage, the stress is constant and these mechanisms lead to decreased strain rates. Secondary stage creep includes increased strain energy stored in the metal because of deformation with the high temperature providing the driving force for the process recovery. Hence there is the balance between work hardening and recovery. Recovery involves the reduction in the dislocation density and the rearrangement of dislocations energy arrays. Dislocations have slip and climb in order that this process combines a good amount of atomic movements should occur, which may also result in diffusion. If the atoms have enough energy to jump into a neighboring site and if there is vacancy at that site in the closed packed lattice, the process is called as self-diffusion. Atoms have more thermal energy as the temperature increases and the equilibrium concentration of the vacanciesin the metal increases exponentially – then, diffusion of metal atom within the metallic crystal will be on larger scale.Some obstacles such as the following, hinder dislocations' movements [3]:

I. Grain boundaries
II. Impurity particles
III. The stress field around solute atoms in the solution
IV. The strain fields of other dislocation

When dislocations cannot move pile up will occur. Figure 2.2 illustrates pile up of dislocations in a crystal.

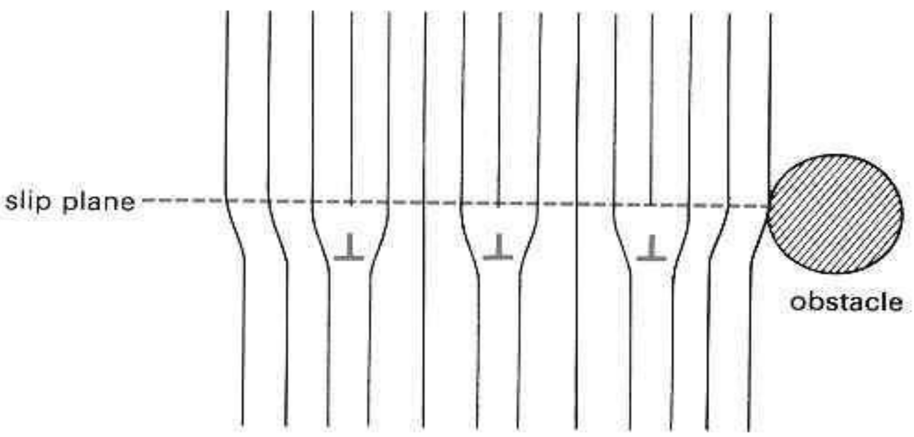

Fig.2.2 Pile up dislocations

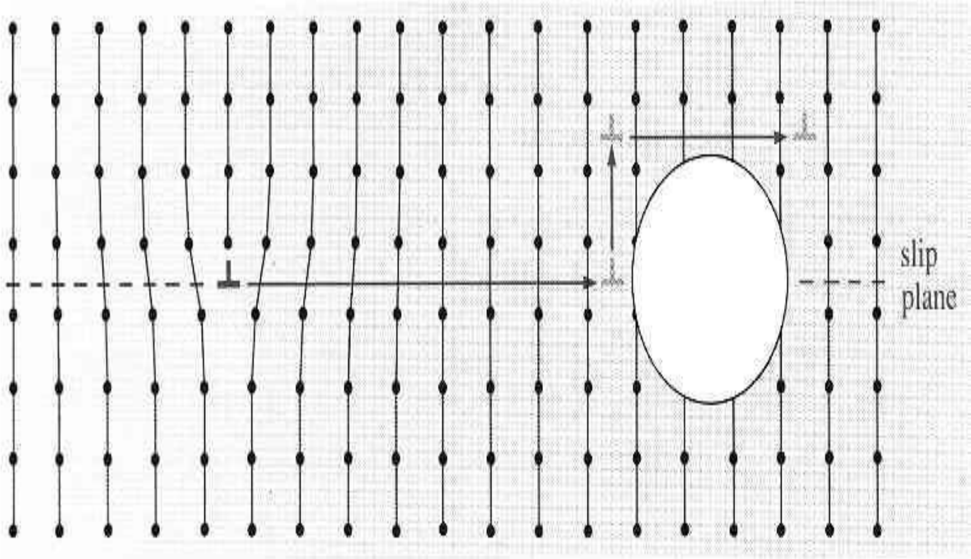

Fig.2.3 Dislocation climbs and slips past an obstacle

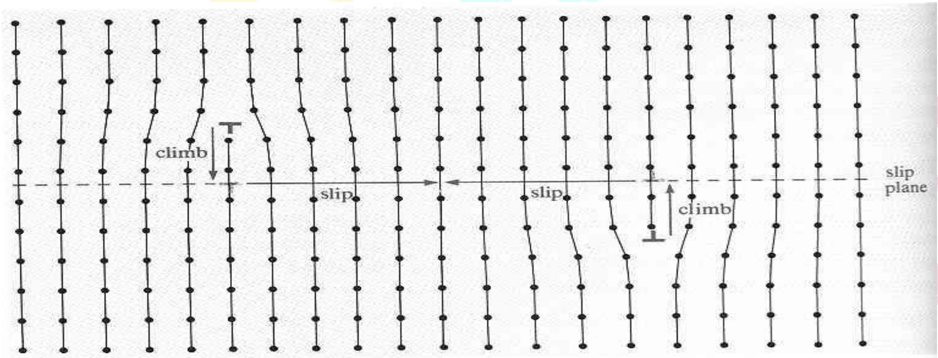

Fig.2.4 Slips to annihilation and opposite sign slip

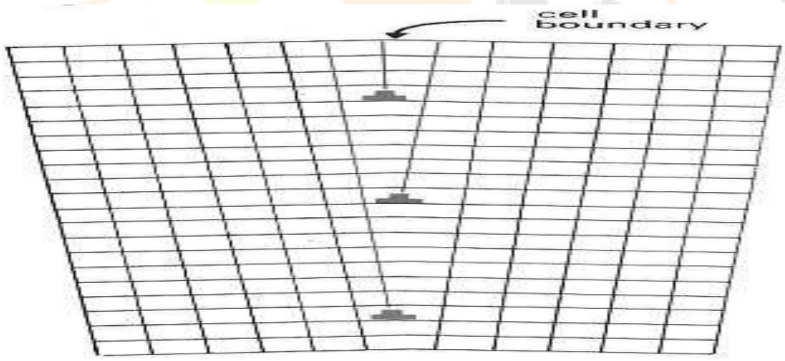

Fig. 2.6 Dislocation wall produced resulting in cell boundary

The jogs are produced when the vacancies diffusion takes place on the bottom of the dislocation but eventually when the line of atoms along the bottom of the dislocation is diffused away, the dislocation will have climbed normal to its slip plane by one atomic spacing. When the dislocation has climbed to an unobstructed slip plane, it will then be free to slip back the obstacle (figure number 2.4 and 2.5). Dislocations of opposite signs attract each other and annihilate each other. By the similar processes dislocations of same sign will align vertically above each other in the dislocation wall. This happens because the stress field around the edge dislocation is compressive above the slip plane and tensile below it to balance out these stress fields and to reduce the total energy interaction. Figure 2.6 depicts formatting dislocation wall resulting in a cell boundary [4].

IV. CREEP IN HETEROGENEOUS MATERIALS

In heterogeneous materials, creep is affected by grain size, microstructure and previous strain history (e.g., cold work) and creep is extremely structure sensitive in these materials. The major factor in creep is grain size. Usually, coarse grained materials exhibit better creep resistance than fine grained ones, since the latter have a higher amount of grain boundary materials and grain boundaries behave as quasi-viscous material with a high tendency to flow at elevated temperatures. This explains thehigher creep resistance of single crystals than polycrystalline materials. The thermal stability of the microstructure of alloys and its resistance to oxidation at high temperatures is another important factor. An annealed specimen for having greater thermal stabilities is far superior in its creep resistance to a quenched steel with its poor thermal stability.If the structural damage happens in an alloy severally, then it is tertiary creep stage, which happens after considerable time of bearing a particular degree of stress and a higher level of temperature in most of the cases. Mainly round and wedge shaped voids are seen in the alloys which when coalesce, creep rupture takes place. When the shear stress acts on the boundaries, the formation of extra voids occur at the grain boundaries- figures 3.1, 3.2 and 3.3 illustrate formation of voids at lateral cracks on grain boundaries so also the phenomenon of grain boundary sliding (GBS) [5].

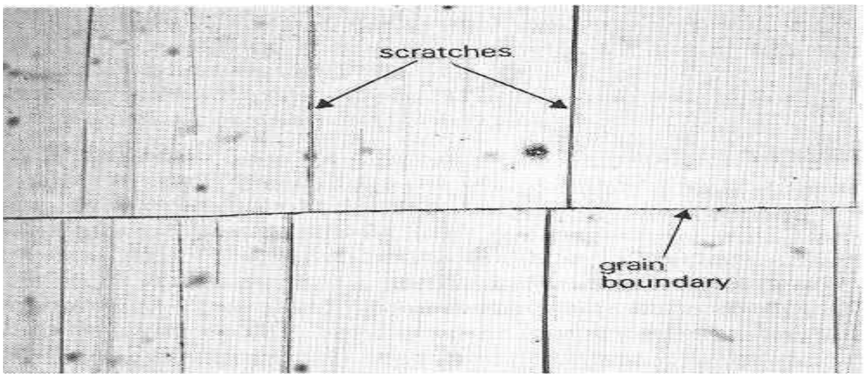

Fig 3.1Grain boundary sliding

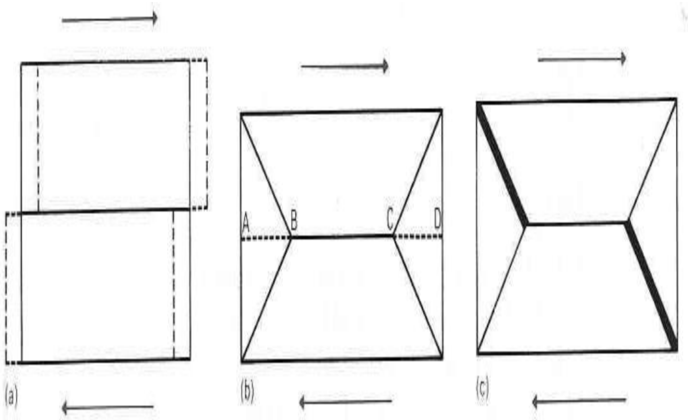

Fig.3.2 Formation of cracks

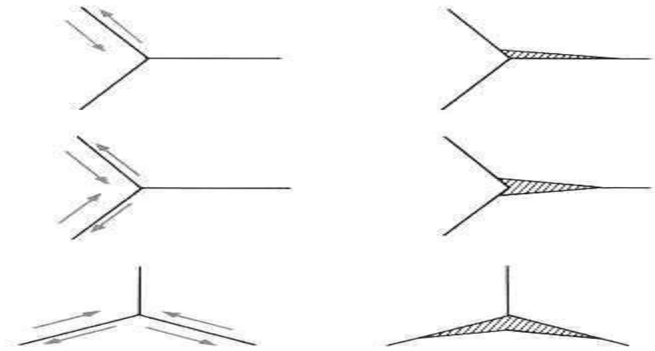

Fig. 3.3Wedge cracks

When the first step on the grain boundary takes place, a square void is generated initially this will turn on to round voids because of the diffusion which reduces the surface area and energy of the void. The grain boundary may influence the total creep from 10 percent to 65 percent. This contribution increases with increasing temperature and stress level. Grain boundary has lower shear strength say at temperature of about 0.6Tm (Tm: Melting point of the material) , because of loose atomic packing at the grain boundaries. Boundaries at 450^{0}C to the applied stress have highest shear strength and will slide the most. Atomic diffusion is also easier and plays a role in contributing to creep. It has been seen that ordering and precipitation dominate at one stage of the creep in materials. Figure 3.4 illustrates boundary diffusion phenomena making the slip difficult and also decreasing the grain boundary strain [5].

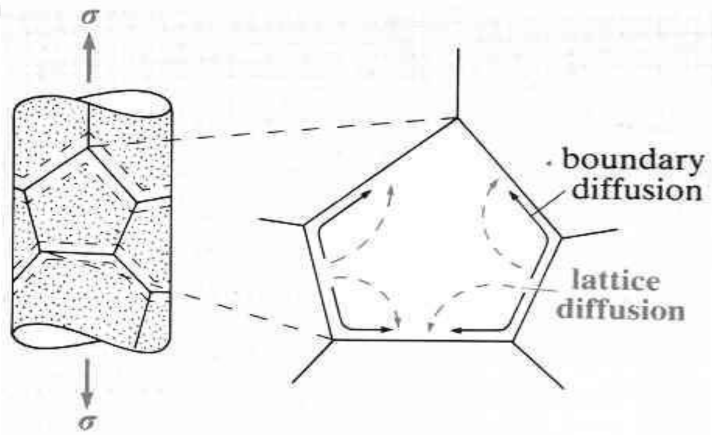

Fig.3.4 Diffusion flow during creep

The creep which takes place at low stress and high temperature is because of diffusion flow in many of the materials. Under the action of driving force the grains diffuse from side to bottom and to top. The grain becomes longer when the applied stress is still constant and the process takes faster at high temperatures as there are more vacancies. The atoms have slower jump frequency for diffusion paths through the grains and this is called Nabaro-Herring creep (N-H Creep). But the jump frequency is higher along the grain boundaries which is called Coble creep [6].

V. MICROSTRUCTURAL OBSERVATIONS AND SCANNING ELECTRON MICROSCOPE IMAGE STUDIES

Fig. 4.1 Microstructure Observed in the Optical Microscope for the Base Metal (Magnification: 500X)

Fig. 4.2 Microstructure Observed in the Optical Microscope for the Weld Metal (Magnification: 500X)

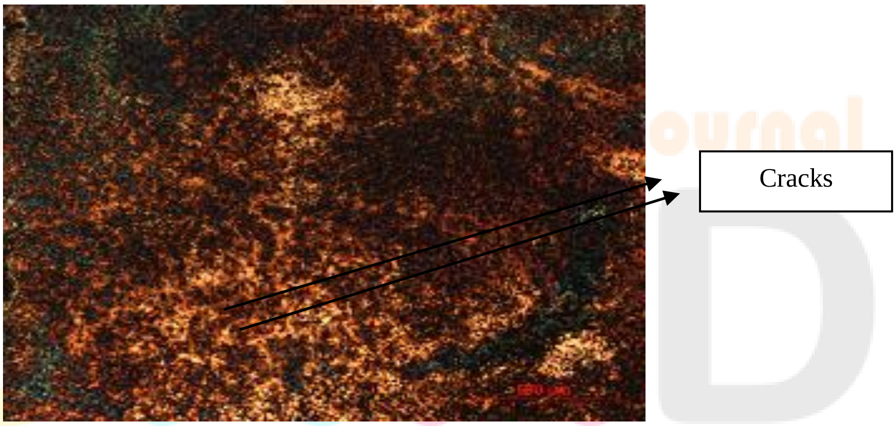

Fig.4.3Microstructure Observed in the Optical Microscope for the Metal taken from HAZ (Magnification:500X)

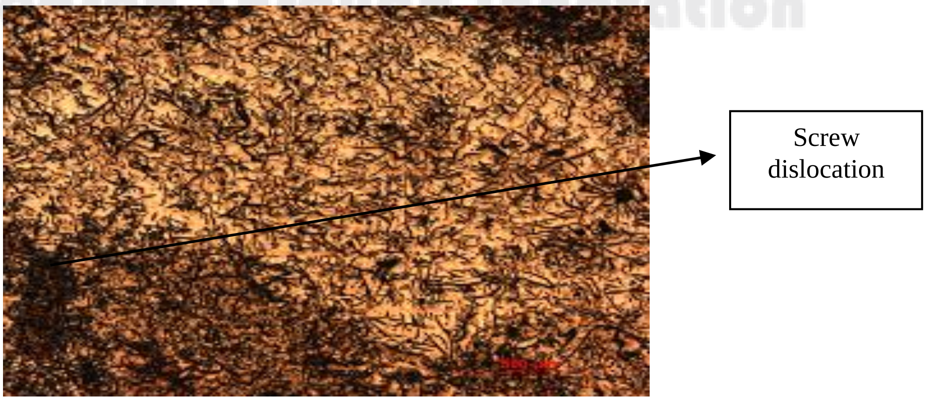

Fig. 4.4 SEM image of the Base Metal (Non-aged, Magnification 500X)

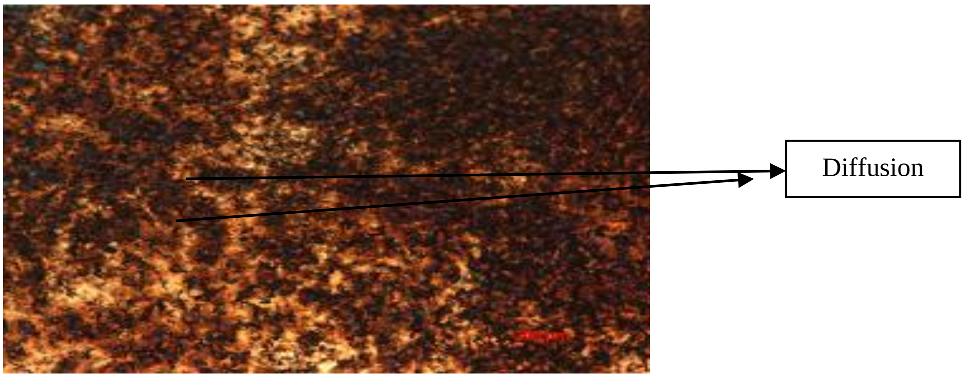

Fig. 4.5 SEM image of the Weld Metal (Non-aged, Magnification 500X)

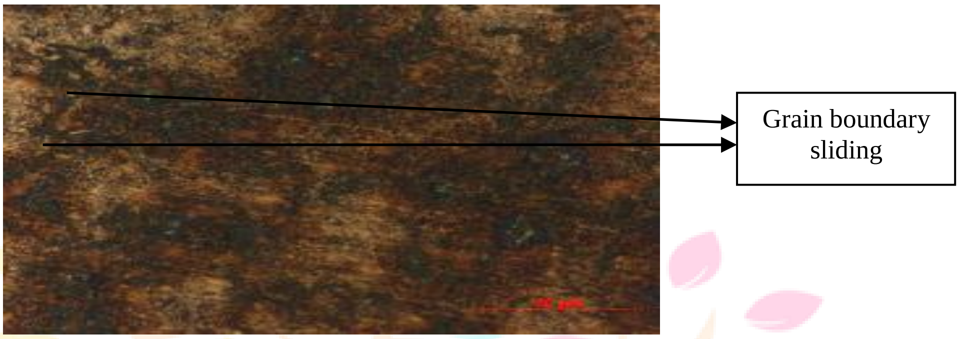

Fig. 4.6 SEM image of the HAZ Metal (Non-aged, Magnification 500X)

VI. MICRO-STRUCTURAL CHARACTERISATION

Grain boundaries separating one crystal from another in a polycrystalline aggregate are structural discontinuities that represent regions of random misfit, ledges and boundary- dislocations. As a result of their structures and high surface energy, grain boundaries influence the mechanical properties of materials. Various studies have revealed that grain boundaries play an important role in the deformation and fracture of polycrystalline materials. At temperatures above 0.5Tm , where Tm is the melting point of the material, this role is reflected in several activities which include: (i) the occurrence of boundary sliding and migration, (ii)the formation of triple-point folds and cavities and (iii) the development of vacancy concentration gradients as a result of tensile and compressive stresses on orthogonal boundaries (diffusion creep). In addition, the presence of boundaries serves as sites for the accumulation of impurities. As a result of this process, referred to as boundary segregation, the impurity concentration at boundaries may be enhanced relative to the matrix by a factor ranging from 10 to 10^3. Over the time, it has been demonstrated that impurity segregation at boundaries can explain many phenomena such as temper embrittlement in steels.From the chemical composition of Grade 91 base metal and weld metal used which is shown in Table 5,it is seen that V and Nb contents are minimum in weld metal whereas Ni is on the higher side of the range. Acidic components in the flux system release oxygen to be in the weld to the extent of 700ppm, which is not measured. The microstructures of the base metals reveal tempered martensite (fig.5.1).

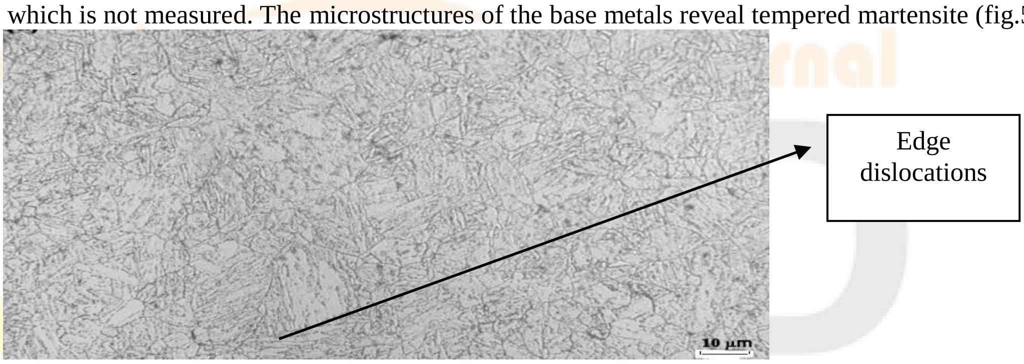

Fig. 5.1. Microstructure of As received 9 Cr-1Mo (Modified) Steel- Optical Micrograph

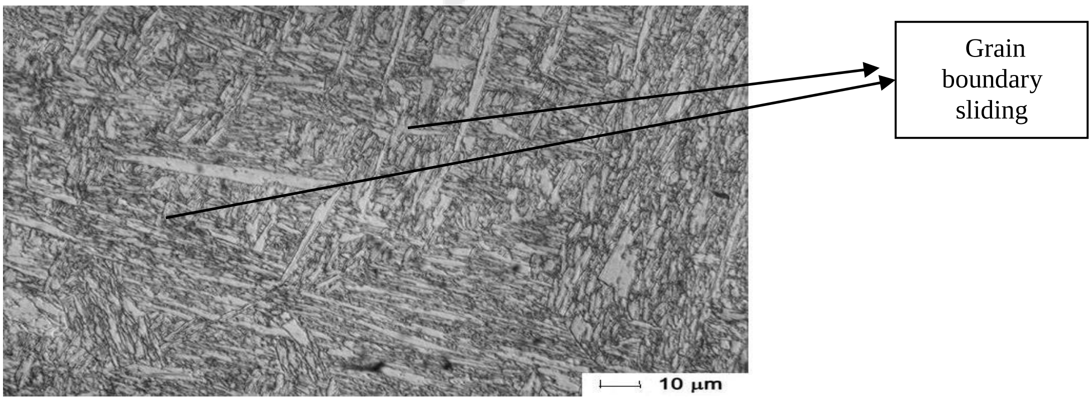

Fig. 5.2. Microstructure of Weld of 9 Cr-1Mo (Modified) Steel- Optical Micrograph

There are some precipitates formed after tempering, which are M23C6 precipitates, as revealed by Energy dispersive X-ray spectroscopy. Spherical micro-inclusions are seen in the optical micrographs of the weld metal (fig.5.2). Micro-inclusions are rich in silicon due to the acidic nature of flux in the coating. During welding, the weld fusion zones solidify as ferrite, but pass through the austenite phase field on cooling; the austenite eventually transforms to martensite well before room temperature is reached. Within the weld of the steel, structures seen represent un-tempered martensite wherein some precipitates are observed in addition to inclusions, both on the prior austenite grain boundaries and along the lath boundaries. A relatively high Ms temperature (420 ^{0}C) because of low carbon content in weld metal, ensures formation of martensite.

The microstructure of the weld metal reveals lath martensitic structure with some needle shaped precipitates present. EDS analysis of precipitates has shown that they are rich in iron, chromium and niobium. During post-weld heat treatment, the martensite gets tempered, carbides and carbo-nitrides precipitate out during tempering. Extent of precipitation, extent to which lath structure gets broken up and extent of sub-grain formation depends upon the degree of ageing or the extent of post-weld heat treatment. Precipitates including those of M23C6 are often on both prior austenite and sub-grain boundaries. It has been reported elsewhere that if V and Nb percentage is high in the welds, precipitates which are rich in these elements are found.

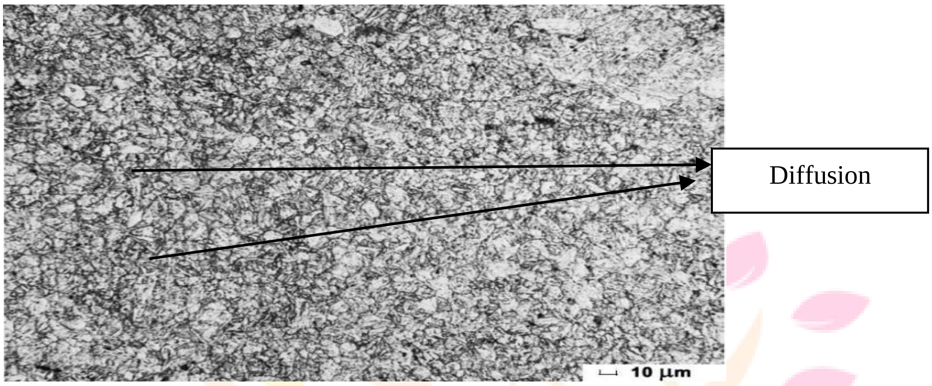

Fig. 5.3. Microstructure of Aged 9 Cr-1Mo (Modified) Steel/ Base Metal- Optical Micrograph-Ageing done for 1000 hours at 700 ^{0}C

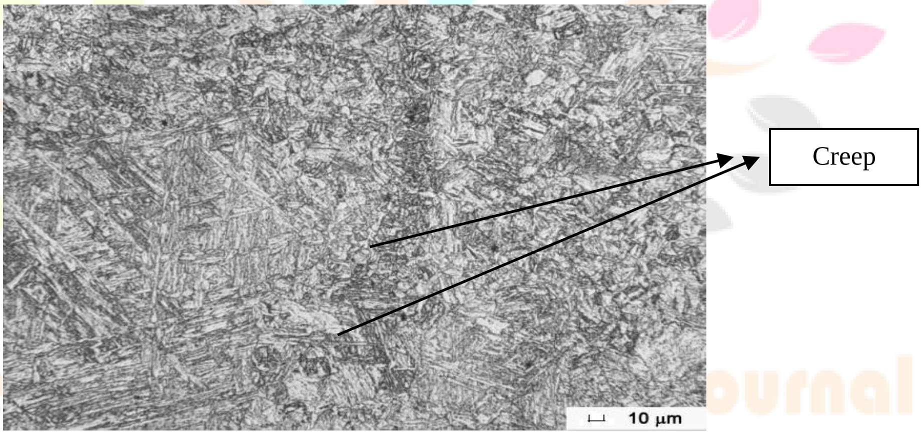

Fig. 5.4 Microstructure of Aged 9 Cr-1Mo (Modified) Steel/ Weld Metal- Optical Micrograph-Ageing done for 1000 hours at 700^{0}C

The increase in the creep values is predominant in the tests conducted at higher temperatures for the aged samples of the steel. It can be concluded that effect of ageing is exhibited at the higher temperature tests for these steels particularly when creep tests are conducted.

VII. RESULTS AND CONCLUSIONS

From figure 4.1 we can identify the point defects easily. When a stress above a critical value is applied to a metal crystal, the crystal lattice slips as dislocations shown in figure 4.2. Screw dislocations can be seen from figures 4.1 and 4.4.Grain boundaries which occur because of high temperatures is showed in the figures 4.6 and 5.2. Weld joint of altered 9Cr-1Mo steel has temperamental microstructure, which is uncovered by changing wet blanket rates exhibited in the space crawl tests led at various stress and temperatures. . Maturing of the steel decays its quality, as uncovered by higher sneak rates seen in the tests led on the matured specimens. Crawl rates are higher for base metal and for the metal removed from the warmth influenced zone (HAZ), contrasted with the weld metal, in instances of low temperature and lower stack tests and of the two, HAZ metal crawls more than the base metal. Nonetheless, when the test load and test temperature are expanded, the weld crawl rate improves and outperforms the crawl rate of base metal and that of HAZ metal. Matured weld tests of changed 9Cr-1Mo steel likewise show higher crawl rates when tried for crawl at higher temperatures and higher stresses.

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