

# Microstructural Investigation of Aged Modified 9Cr-1Mo Steel Welded Joints Correlating Creep Behaviour

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## Abstract

There has been a constant search for new materials for high temperature applications, such as for parts of steam generators, parts of thermal power plants, piping of liquid metal reactors, which are likely to be exposed to temperature 550°C and above. Good strength and ductility values possessed by special steels make them the first choice of designers for applications mentioned. Modified 9Cr-1Mo steel has been in use for such applications since a decade. For any given material, along with the mechanical properties, there is a need to determine the creep property if the material is the choice for high temperature service applications. Further, as it has been experienced that the welds are likely zones of failure, under extreme conditions of load and temperature, probing the creep property of the welded joints of a given material becomes an important aspect of research. Hence it is proposed to probe into the creep resistance aspect of the welded joints of 9Cr-1Mo steel under different conditions of load and temperature. It is also proposed to age the samples of 9Cr-1Mo steel under different conditions so that variation of creep properties in both the parent and the weld metal can be studied adopting the indentation creep test methodology. Experiments will be designed to extract all possible information regarding structural disintegration of the material. Additionally, scanning electron microscopy is planned to be adopted to observe micro-structural changes and correlation will be attempted to link them to observed creep behaviour.

**Keywords:** 9Cr-1Mo, Scanning Electron-Microscope, Martensitic Matrix, Creep behavior, Indentation Creep

## I. INTRODUCTION

Vital efforts are needed to determine the creep property if the material is the choice for high temperature service applications. Further, as it has been experienced that the welds are likely zones of failure, under extreme conditions of load and temperature, probing the creep property of the welded joints of a given material becomes an important aspect of research.[3] Hence it is proposed to probe into the creep resistance aspect of the welded joints of 9Cr-1Mo steel under different conditions of load and temperature.[1] While tensile creep tests are time consuming and cumbersome, the indentation creep tests can help the investigator to probe into the creep property of the material in a shorter time. Indentation or Impression creep tests, as they are popularly called, refer to tracking of time dependent motion of an indenter into a solid under constant load at a given temperature. Under each test, strain experienced by the sample is plotted against time, to obtain the creep curve which is known to have one to one correspondence with a tensile test creep curve. Temperature and loads for the test may vary to simulate practical conditions. They have also made an effort to correlate structural factors like dislocation density, grain size and nature of grain boundaries to the deformation pattern under the punch. The advantage of this methodology is that a small specimen is enough to assess the creep properties, thereby allowing the investigator to study the local variation in properties of a larger sample. Experiments are designed to extract all information regarding structural disintegration of the material. Then, scanning electron microscopy is adopted to observe micro-structural changes.[10]

## II. GENERAL 9 CR STEEL GRADES

Creep resistant 9-12% Cr steels were originally developed for gas turbine applications in the 50's.[2] Additions of elements like molybdenum (Mo), vanadium (V) and niobium (Nb) were practised during subsequent development of this group of alloys which showed high hardenability due to the high content of alloying elements so that, after heat treatment, a martensitic microstructure was formed even in large sections at relatively low cooling rates. In the mid of 70's the 9 Cr 1Mo alloy, used for tubing in the UK was further modified by material scientists with the addition of V, Nb and nitrogen (N) and was included in the ASTM standard as Grade 91.[2]

Table - 1

The chemical analysis of modified 9 Cr-1 Mo steel and its comparison with standard 9 Cr-1 Mo steel [2]

Chemical elements	Content Range, wt %	
	Modified 9 Cr-1 Mo [Grade 91]	Standard 9 Cr-1 Mo (Grade 9)
Carbon	0.08-0.12	0.15 max
Manganese	0.30-0.60	0.30-0.60
Phosphorus	0.020 max	0.030 max
Sulphur	0.010 max	0.030 max
Silicon	0.20-0.50	1.00 max
Chromium	8.00-9.50	8.00-10.00
Molybdenum	0.85-1.05	0.90-1.10
Nickel	0.40 max	
Vanadium	0.18-0.25	
Niobium	0.06-0.10	
Nitrogen	0.030-0.070	
Aluminium	max	

### III. CREEP IN STEELS AND IN GRADE 91 STEEL

The progressive deformation of a material at constant stress is called Creep. Despite the material being deformed plastically at ambient temperature, work hardening increases the strength of the material. During work hardening, prior dislocations obstruct the passage of any new dislocations, thus increasing the strength of the material. But at elevated homologous temperatures due to thermal energy, the dislocations annihilate and the work hardening is weakened. Creep deformation and rupture of metals is of great concern for prolonged service life at elevated temperatures. For an engineering structure operating in high temperature, creep is not considered as a significant deformation mechanism. In a high temperature application, parameter like the duration of test, the grain size, sub grain size, precipitate size, effective stress all have a role in dictating the strength of the material.[2] A typical & conventional creep curve consists of three distinct stages: primary, secondary and tertiary. In primary creep, the strain rate (or creep rate) decreases with time as work hardening takes the center stage. The secondary stage, also known as steady-state creep, is of vital importance as it sheds light on the creep deformation mechanism. In the secondary creep stage, the material is deformed plastically while there is a higher degree of strain hardening and increased dislocation density, resulting in a constant creep rate. Strain rate increases as time increase in tertiary creep. . Increased creep rate results in necking, crack, and void formation. Tertiary creep stage is very rapid so the materials in engineering structures are designed not to enter this stage. [2]

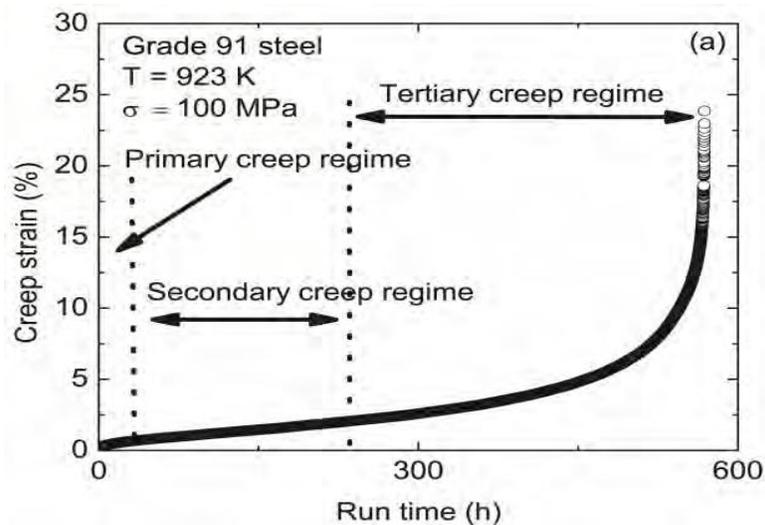


Fig. 1: (a) A typical creep curve [2]

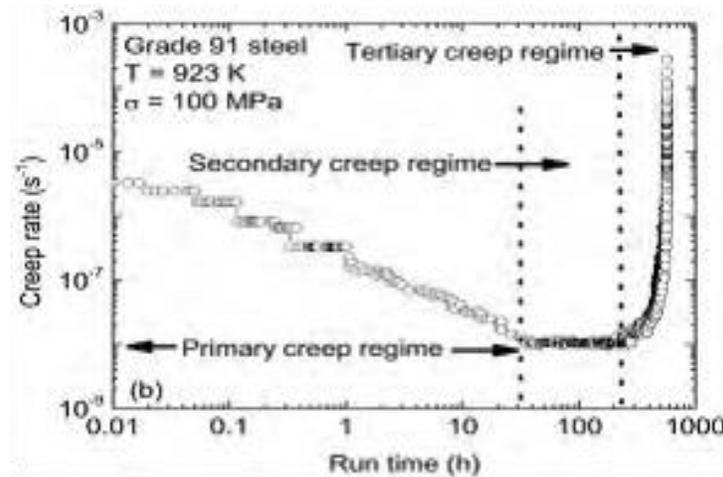


Fig. 1: (b) creep rate vs. time showing three distinct stages. [2]

#### IV. WELD CHARACTERISTICS OF GRADE 91 STEEL

Welding of Grade 91 steel creates a heat affected zone (HAZ) resulting in changes in microstructure because of the temperature gradient during welding. As-welded and post weld heat treated (PWHT) specimens have weld zone, unaffected base material (BM) and HAZ. HAZ has three distinct microstructures, i.e. coarse, fine grain and partially austenitized grain structures and an indistinctive columnar structure formed after directional solidification in the weld zone, as shown in figure. [2]

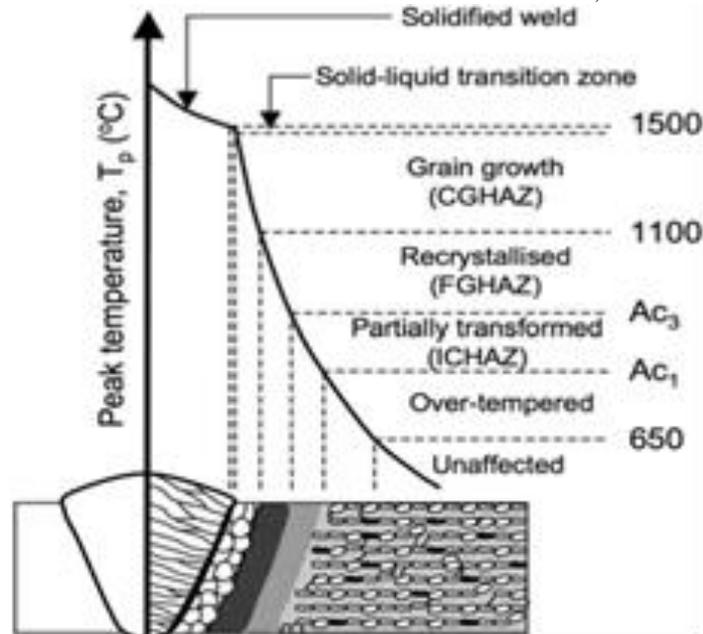


Fig. 2: Schematic representation of microstructures developed in welded 9Cr-1Mo steel as approximate function of peak temperature during welding. [2]

#### V. TEST SPECIMENS



Fig. 3: a) as received specimen



Fig. 3: b) After welding



Fig. 4: a) Before indentation creep test



Fig. 4: b) After indentation creep test

## VI. RESULTS

### A. Creep Results:

Table – 1  
Steady State Creep Rates (SSCRs) for 9Cr-1 Mo Samples [8 Kg Load]

Sample Type	SSCR values in mm/mm/min	
	Room Temp test	Test at 3500C
Base Metal	$6.086 \times 10^{-5}$	$8.1 \times 10^{-5}$
Weld Metal	$5.96 \times 10^{-5}$	$7.62 \times 10^{-5}$
HAZ area Metal	$6.10 \times 10^{-5}$	$8.25 \times 10^{-5}$

### B. Mechanical Results:

Table – 2  
Yield Strength (YS) values and the Ultimate Tensile Strength (UTS) values for Non-Aged 9Cr-1 Mo Samples

	Yield Strength, MPa	Ultimate Tensile Strength, MPa
Base Metal	405	579
Weld Metal	408	566
HAZ area Metal	387	548

Table - 3  
Yield Strength (YS) values and the Ultimate Tensile Strength (UTS) values for Aged 9Cr-1 Mo Samples [10]

	Yield Strength, MPa	Ultimate Tensile Strength, MPa
Base Metal	409	557
Weld Metal	422	532
HAZ area Metal	403	540

## VII. MICROSTRUCTURAL OBSERVATIONS AND SCANNING ELECTRON MICROSCOPE IMAGE STUDIES



Fig. 5.1: Microstructure Observed in the Optical Microscope for the Base Metal (Magnification: 500X)



Fig. 5.2: Microstructure Observed in the Optical Microscope for the Weld Metal (Magnification: 500X)



Fig. 5.3: Microstructure Observed in the Optical Microscope for the Metal taken from HAZ (Magnification: 500X)

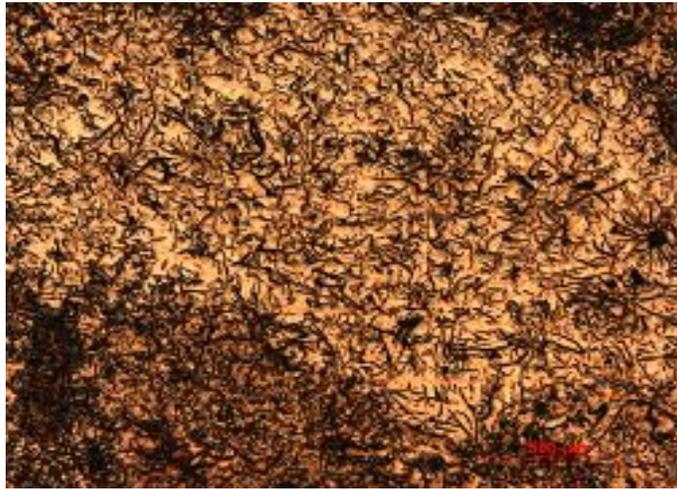


Fig. 5.4: SEM image of the Base Metal (Non-aged, Magnification 500X)

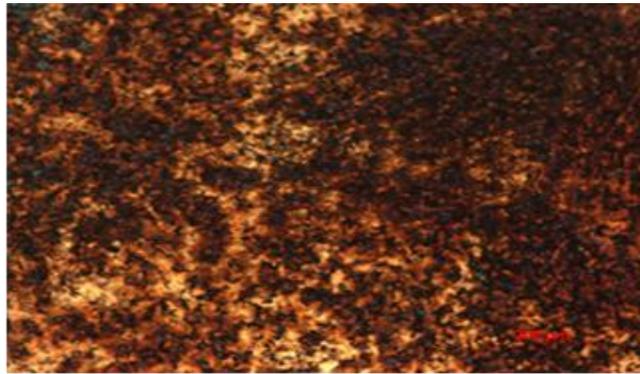


Fig. 5.5: SEM image of the Weld Metal (Non-aged, Magnification 500X)



Fig. 5.6: SEM image of the HAZ Metal (Non-aged, Magnification 500X)

### VIII. DISCUSSION

It can be observed that there is variation in the mechanical strength values across base metal, weld metal and the region of HAZ. SSCR values recorded are higher for the parent metal compared to the weld metal and the values recorded for the HAZ. This depicts a better situation for the weld metal, microstructural observations indicate that weld metal is not homogeneous and further probing regarding its creep resistance is needed.

### IX. CONCLUSION

Microstructure of the Weld metal is heterogeneous; ageing treatment further modifying it. Creep rate is more for Base Metal and for the metal taken out of the heat affected zone (HAZ) compared to the weld metal and of the two, HAZ metal creeps more than the Base Metal. The statement holds good for higher temperature tests also. The creep properties did not significantly change because of the ageing treatment. As all the samples are normalized (at 1050 C) and tempered (at 750C), martensitic structure is depicted that provides good carbide and nitride solubility. The desired microstructure with tempered martensitic matrix with V and Nb carbo-nitrides and Cr carbides is almost achieved, as can be seen.

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