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## **Metallurgical Investigation of Material from Chill-Water Piping System**

D. J. Alexander

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METALLURGICAL INVESTIGATION OF MATERIAL  
FROM CHILL-WATER PIPING SYSTEM

D. J. Alexander

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## CONTENTS

	Page
LIST OF FIGURES . . . . .	v
ABSTRACT . . . . .	1
INTRODUCTION . . . . .	1
EXPERIMENTAL PROCEDURE . . . . .	2
RESULTS . . . . .	4
DISCUSSION . . . . .	11
CONCLUSIONS . . . . .	15



## LIST OF FIGURES

Figure		Page
1	Schematic of pipe shows orientation of test specimens and sections used for examination of microstructure . . . .	3
2	Unetched microstructures show inclusion size and distribution. Left, pipe L; right, pipe S . . . . .	5
3	Etched microstructures show grain size and pearlite distribution. Left, pipe L; right, pipe S . . . . .	6
4	Effect of temperature on yield and ultimate strengths of pipe materials . . . . .	8
5	Effect of temperature on ductility of pipe materials . . .	9
6	Charpy V-notch transition curves for pipe materials . . . .	10
7	Fracture surfaces from specimens tested at low temperatures . . . . .	12
8	Fracture surfaces from specimens fractured at higher temperatures . . . . .	13



METALLURGICAL INVESTIGATION OF MATERIAL  
FROM CHILL-WATER PIPING SYSTEM\*

D. J. Alexander

ABSTRACT

The mechanical properties and microstructures of two steel pipes that were removed from the Oak Ridge National Laboratory (ORNL) chill-water system have been studied. Concerns for low-stress failure of aging pipes prompted a metallurgical investigation to determine the risk of using a cryogenic freeze-plug technique to isolate a section of piping for repair. The two pipes, designated S and L, were low-alloy carbon steel, with microstructures of ferrite and pearlite. Pipe S had a small grain size and a banded microstructure, whereas pipe L had a larger grain size with less pearlite, which was randomly spaced. Pipe S had a ductile-to-brittle transition temperature (DBTT) of 0°C, compared to 84°C for pipe L. Because of the high transition temperature and the very low level of the lower shelf, cooling to liquid-nitrogen temperature resulted in a very small margin of safety for these pipes. Therefore, this technique is not recommended for the pipe materials, and caution is advised in applying this technique to any pipe with unknown toughness properties.

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INTRODUCTION

Four sections of piping in the chill-water system at ORNL were removed from service during a maintenance operation to replace a damaged valve. Removal of the sections required shutting down the system and draining the ethylene glycol coolant that was carried by the pipes. An alternative plan that would have allowed replacing the valve without draining the system was suggested. This method would have involved wrapping a jacket around the pipe and cooling it with liquid nitrogen

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\*Based on work performed at Oak Ridge National Laboratory, operated for the U.S. Department of Energy under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

until the ethylene glycol formed a solid plug inside the pipe. The plug would seal the system, allowing the pipe to be cut and the valve to be removed. The repair would be made by welding a new section into the pipe while maintaining the seal with the liquid-nitrogen-cooled jacket. However, it was decided that this method would not be used because of concerns about the toughness of the piping at the very low temperatures that would be required.

This report describes the mechanical testing and metallurgical analyses of the material removed from service. The results of these tests will help to form the basis for a decision concerning the use of the jacket-cooling technique in the event of a similar situation.

#### EXPERIMENTAL PROCEDURE

Only two of the four sections of pipe that were removed from service were thick enough to permit full-size Charpy specimens to be machined. Therefore, only these two pipes were examined. Material was removed from the two pipes, and Charpy and tensile specimens were machined. The Charpy specimens were notched to represent a through-thickness crack running around the pipe diameter. Figure 1 shows the orientation of the test specimens with respect to the pipe dimensions and the orientation of the sections used to examine the microstructure. The pipe with the larger wall thickness was designated L, and the smaller wall thickness was designated S. Pipe L was approximately 216-mm long (8.5 in.) by 222-mm outside diameter (8.75 in.), with a wall thickness of 16 mm (0.625 in.). Pipe S was approximately 216-mm long by 267-mm outside diameter (10.5 in.), with a wall thickness of 13 mm (0.5 in.).

Charpy V-notch (CVN) impact testing was used to determine the ductile-to-brittle transition temperature (DBTT) of the two steels. The impact energy data were fitted with a hyperbolic tangent function to determine the transition temperature, which was defined as the temperature midway between the upper and lower shelves of the fit to the Charpy data. The temperature at energy levels of 40.7 and 67.8 J (30 and 50 ft-lb, respectively) was also determined. Pieces of the

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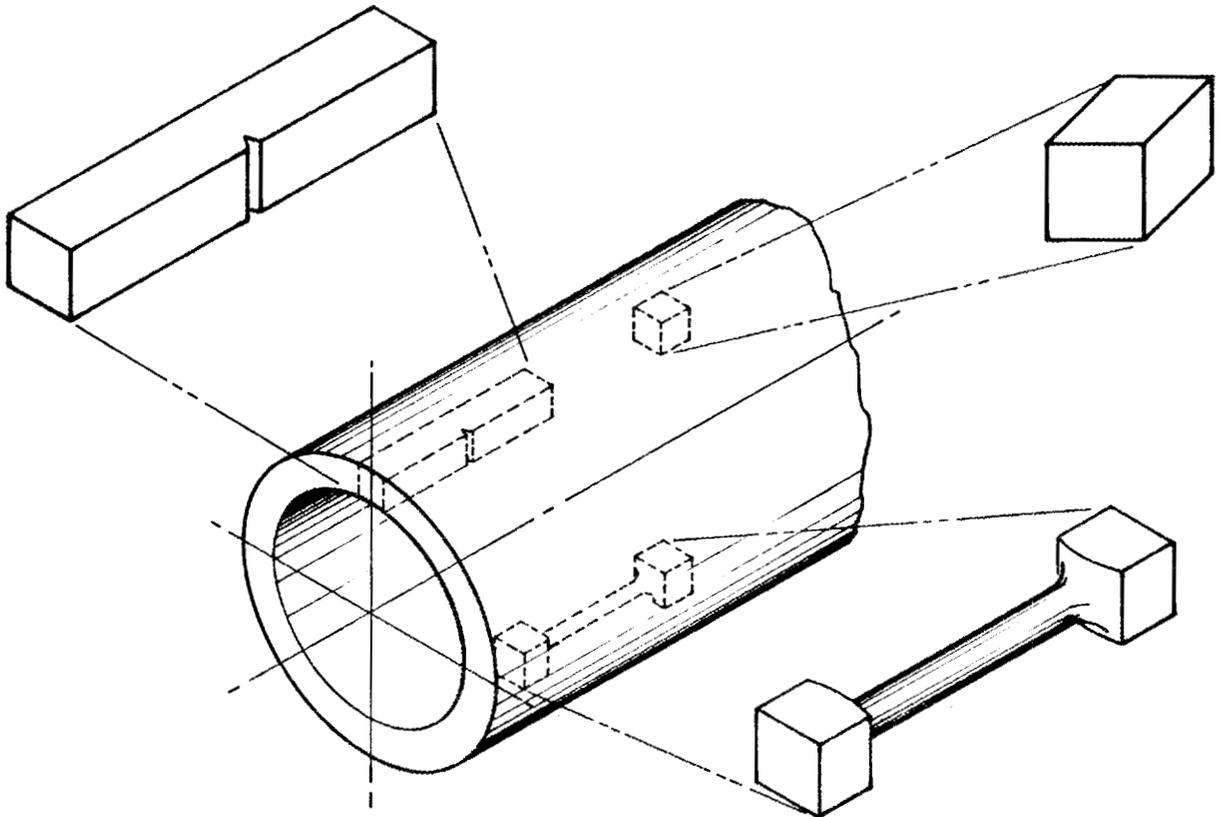


Fig. 1. Schematic of pipe shows orientation of test specimens and sections used for examination of microstructure.

fractured samples were used for chemical analysis and sectioned and polished for metallographic examination. Optical photographs of the metallographic samples were used to measure the volume percent of ferrite by point counting and the linear intercept grain size. Fracture surfaces from specimens tested at both high and low temperatures were examined in the scanning electron microscope.

Tensile testing was conducted at room temperature and at  $-75$  and  $-196^{\circ}\text{C}$ . At  $-75^{\circ}\text{C}$  the specimens were tested in an insulated chamber and cooled with liquid-nitrogen vapor, and at  $-196^{\circ}\text{C}$  the test specimens were immersed in liquid nitrogen. The initial crosshead speed was  $8.5 \times 10^{-3}$  mm/s (0.02 in. min), which corresponded to an initial strain rate of  $2.7 \times 10^{-4}$  s $^{-1}$ .

## RESULTS

The composition of the two alloys is shown in Table 1. The alloys are both low-carbon steels, with pipe S having a greater carbon content.

The microstructures of the two pipes were quite different (Figs. 2 and 3). Pipe L had a larger grain size, with an average linear intercept distance of  $49 \mu\text{m}$ ; pipe S had a much smaller grain size, with an average linear intercept distance of  $17 \mu\text{m}$ . These linear intercept distances correspond to ASTM grain sizes of 5.5 and 8.5, respectively. The microstructure in both cases consisted of ferrite and pearlite grains. The pearlite in pipe L was distributed fairly randomly, whereas pipe S had a banded structure. Also, the steel in pipe S contained a much greater volume fraction of pearlite (measured at 25%), which is

Table 1. Chemical composition of the pipes

Pipe	Element (wt %)							
	C	Mn	Si	S	P	Ni	Mo	Cr
S	0.267	0.73	0.18	0.013	0.014	0.20	<0.05	<0.5
L	0.066	0.43	0.06	0.033	0.087	0.12	<0.05	<0.5

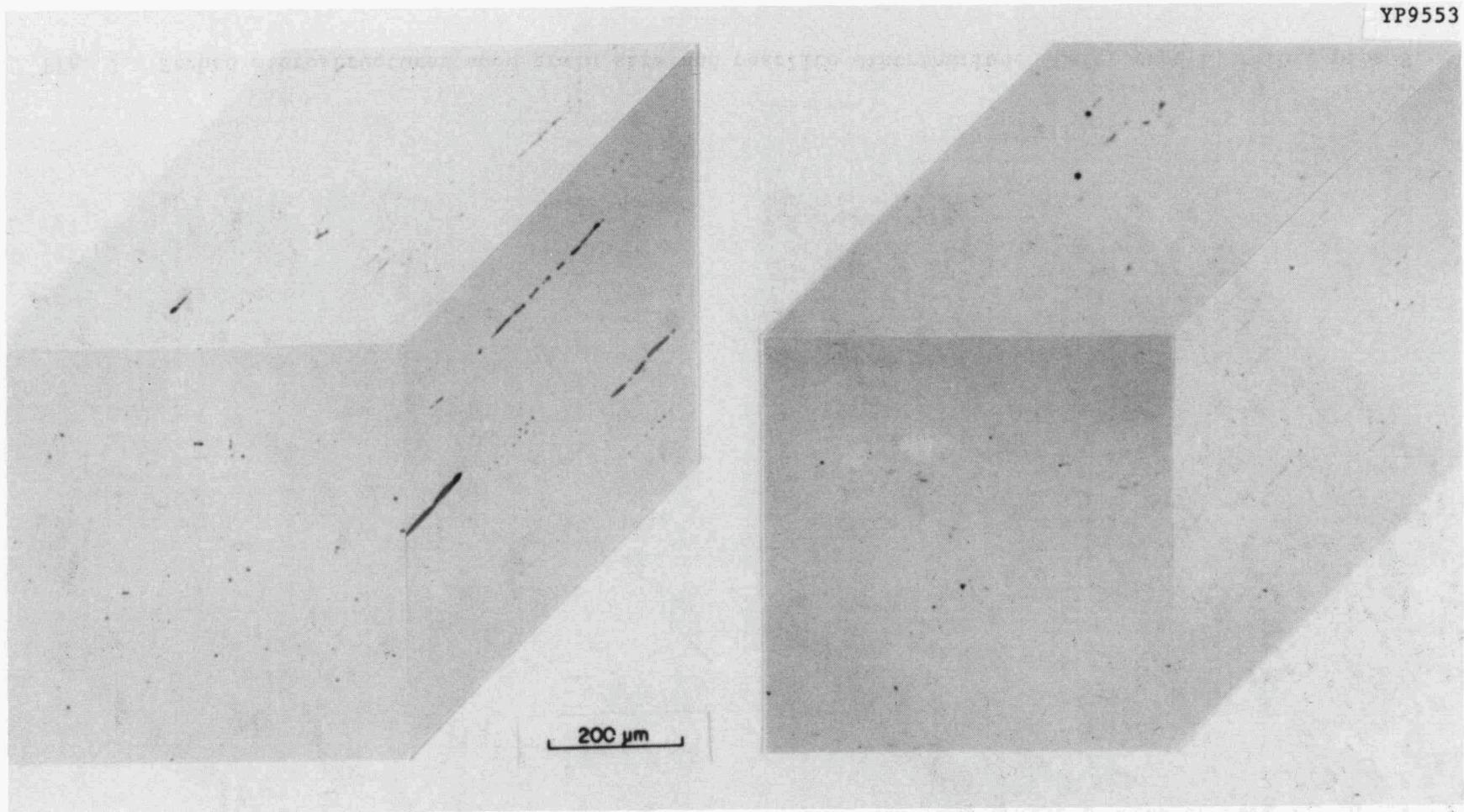


Fig. 2. Unetched microstructures show inclusion size and distribution. Left, pipe L; right, pipe S.

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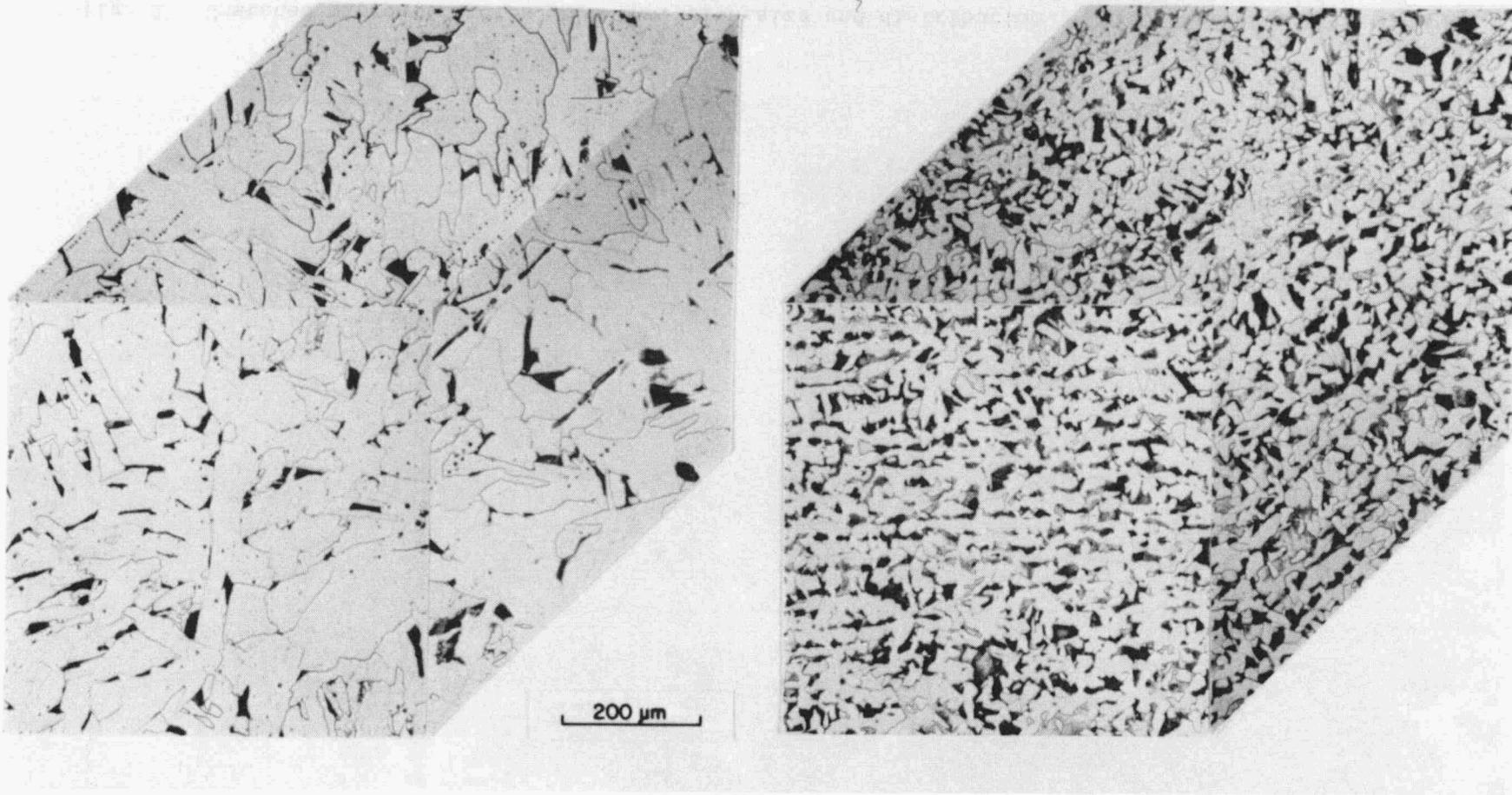


Fig. 3. Etched microstructures show grain size and pearlite distribution. Left, pipe L; right, pipe S.

consistent with its greater carbon content (Table 1). Pipe L had a measured ferrite content of only 5% and contained a greater number of inclusions (Fig. 2). This reflects pipe L's greater sulfur and phosphorus contents, which were evident from the chemical analysis (Table 1).

The tensile properties are given in Table 2 and are shown in Figs. 4 and 5. Pipe S, which had the smaller grain size, had greater yield, greater ultimate strengths, and greater hardness than pipe L. The decrease in test temperature resulted in an increase in the yield and ultimate strengths for both steels. The ductility increased slightly at  $-75^{\circ}\text{C}$  and then decreased significantly at  $-196^{\circ}\text{C}$ . The pipe L material fractured with nearly zero ductility at  $-196^{\circ}\text{C}$ , which reduced the expected increase in the yield and ultimate strength.

Table 2. Mechanical properties of the pipes

Pipe	Yield stress (MPa) <sup>a</sup>	Reduction of area <sup>a</sup> (%)	Hardness ( $R_B$ ) <sup>a</sup>	DBTT ( $^{\circ}\text{C}$ )	Upper-shelf energy (J)	$T_{40.7}$ ( $^{\circ}\text{C}$ )	$T_{67.8}$ ( $^{\circ}\text{C}$ )
L	208	70	62	84	272	72	77
S	355	59	78	0	107	-8	7

<sup>a</sup>Room temperature.

Table 2 summarizes the Charpy impact results shown in Fig. 6. Both pipes displayed the expected transition from ductile to brittle behavior with decreasing temperatures. Pipe S had a lower transition temperature ( $0^{\circ}\text{C}$ ) and a much lower upper-shelf level (107 J) than pipe L, which had a transition temperature of  $84^{\circ}\text{C}$  and an upper-shelf level of 272 J. The latter energy level is an estimate only because no specimens were tested at temperatures high enough to confirm the upper shelf. However, specimens tested at  $100^{\circ}\text{C}$  showed 100% ductile fracture, which should provide a good estimate of the true upper-shelf energy level.

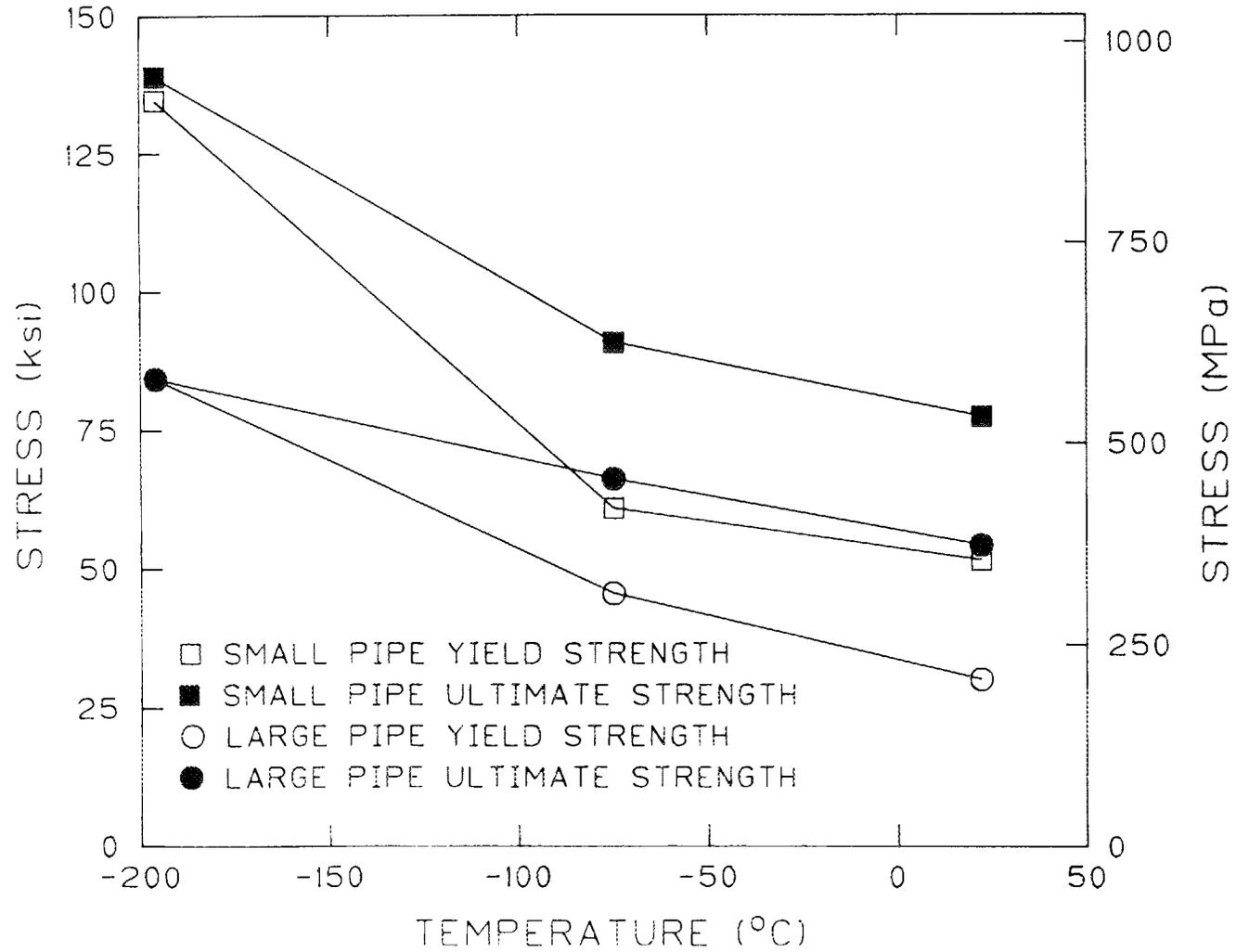


Fig. 4. Effect of temperature on yield and ultimate strengths of pipe materials.

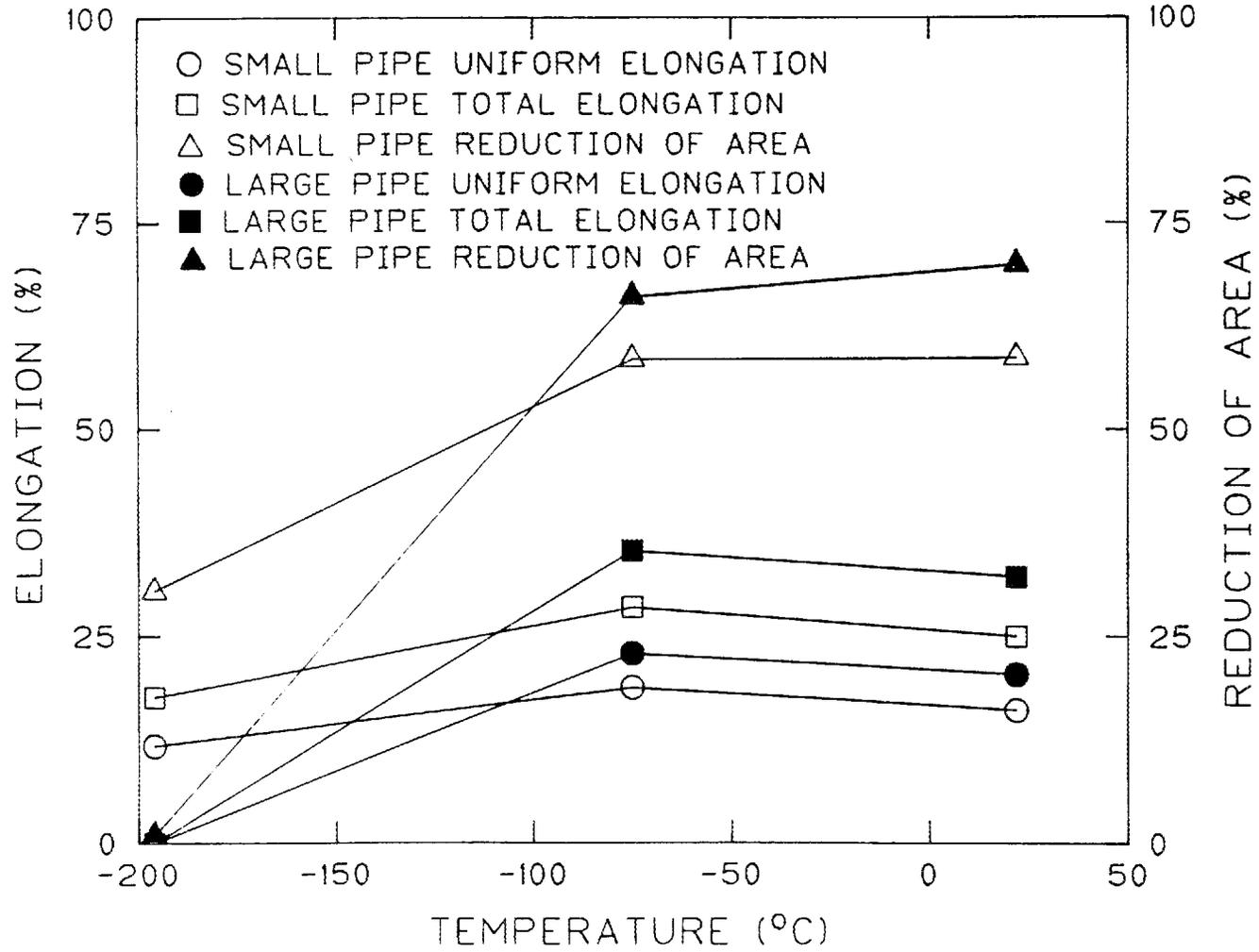


Fig. 5. Effect of temperature on ductility of pipe materials.

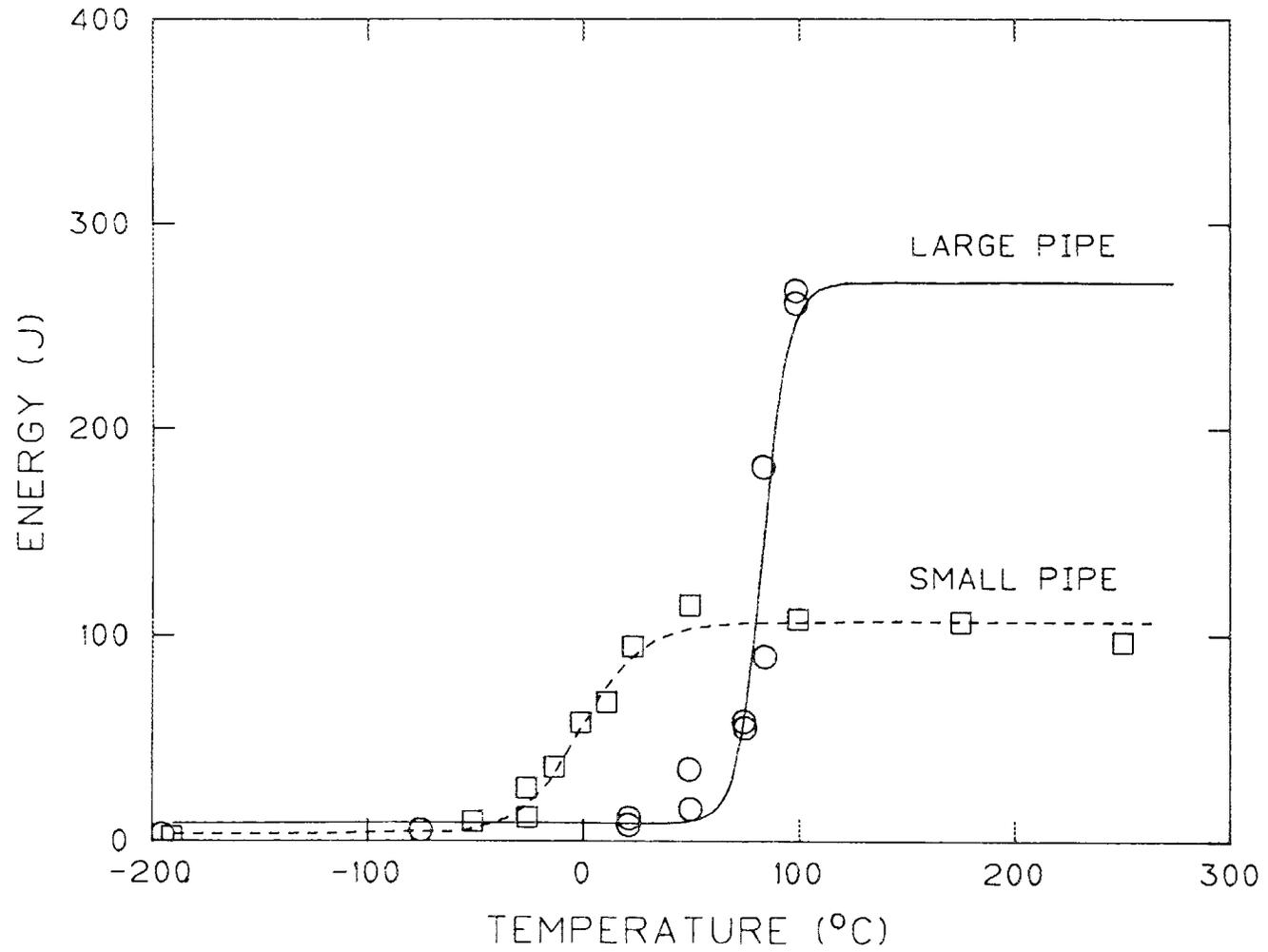


Fig. 6. Charpy V-notch transition curves for pipe materials.

The fracture surfaces are shown in Figs. 7 and 8. Figure 7 shows the fracture surfaces of specimens tested at low temperatures where the fractures were primarily cleavage. The difference in the grain sizes is reflected in the scale of the cleavage fracture facets. The facets from pipe L are much larger than those of pipe S (Fig. 7). Figure 8 shows the fracture morphology of specimens tested at higher temperatures where the fracture mode was ductile tearing. The large grain material typically has smaller dimples than the small grain material, which is consistent with the higher energy levels absorbed on the upper shelf.

### DISCUSSION

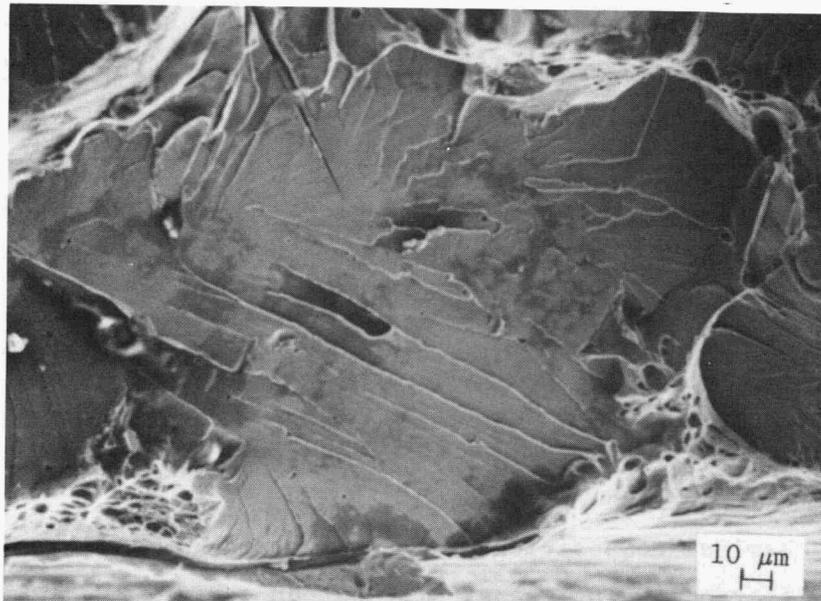
The chemical composition of the two pipes is similar, with the primary difference being the greater carbon content in pipe S. This is reflected in the higher volume fraction of pearlite that is present in pipe S. In addition, pipe S has a much finer grain size. These two factors account for the differences in the mechanical properties.

The yield strength of these steels is mainly a function of the grain size. The finer grain size of pipe S results in greater strength. In addition, the higher volume fraction of pearlite also contributes to an increase in yield strength. However, this increase in the amount of pearlite reduces the ductility of pipe S near room temperature, so pipe L is more ductile, despite its larger grain size. This situation changes when temperatures are quite low and the fracture process changes from a ductile to a cleavage mode.

An increase in yield strength and a decrease in ductility are expected for these ferritic materials as the temperature decreases.

The Charpy impact properties are very interesting. Pipe S has a lower transition temperature but a much lower upper-shelf energy than does pipe L. The lower transition temperature is the result of the finer grain size, whereas the decrease in the upper shelf is caused by the greater volume fraction of pearlite present in pipe S. The finer grain size in pipe S results in smaller fracture facets during the cleavage process at low temperatures. The difference in the size of the fracture facets can be readily seen on the fracture surfaces of the

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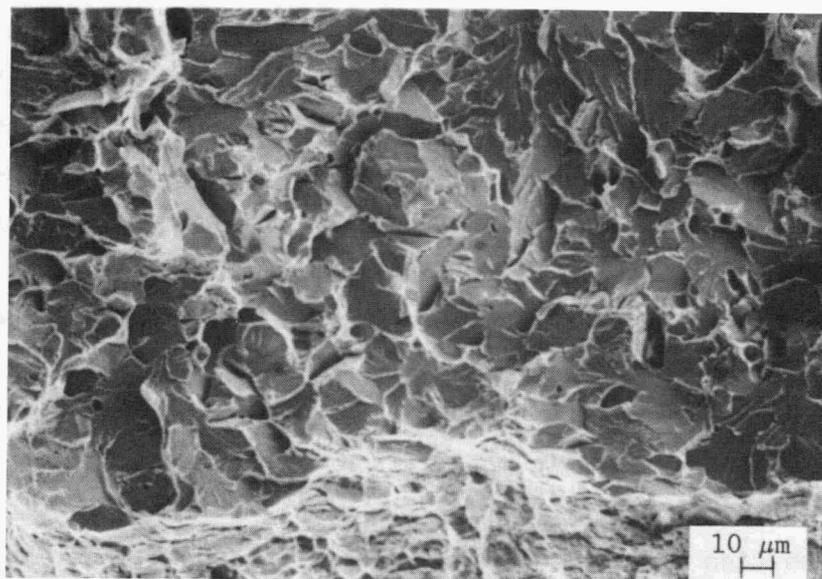
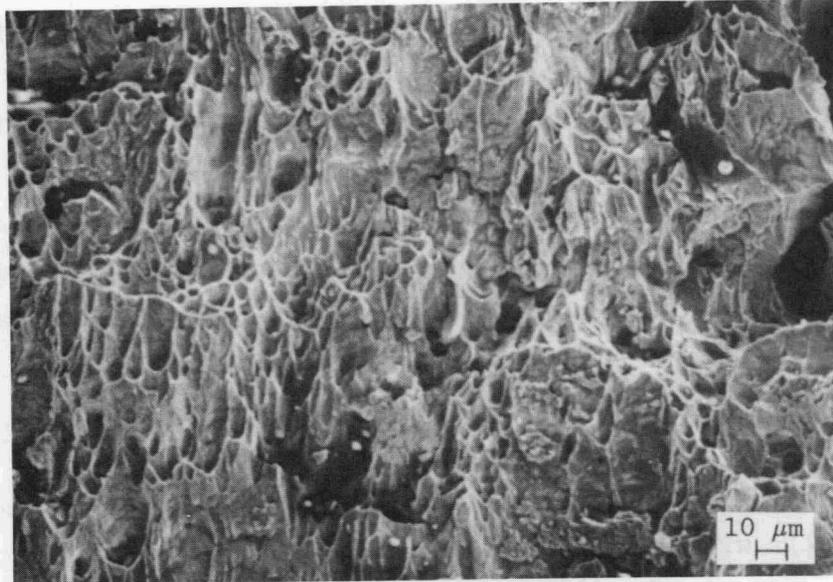


Fig. 7. Fracture surfaces from specimens tested at low temperatures. The notch root is at the bottom of each photograph, with crack growth from the bottom to the top. Top: pipe L, 22°C; bottom: pipe S, -50°C. Note the much larger fracture facets for the large-grain material from pipe L.

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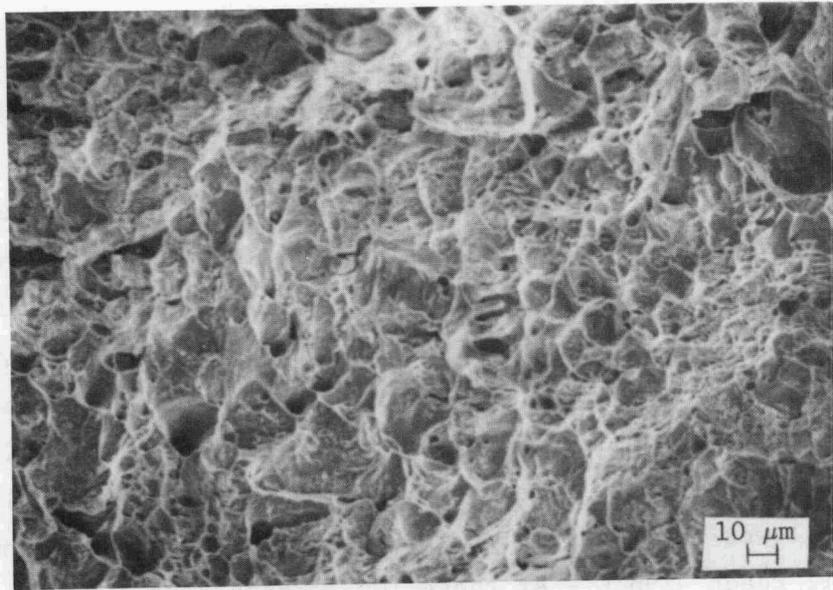


Fig. 8. Fracture surfaces from specimens fractured at higher temperatures. The notch root is at the bottom of each photograph, with crack growth from the bottom to the top. Top: pipe L, 85°C; bottom: pipe S, 100°C.

Charpy specimens. Pipe L produces much larger fracture facets than does pipe S (Fig. 7). The steep slope of the transition region for pipe L is reflective of its lower carbon content.

The difference in the upper-shelf energies is the result of the pearlite content. Pipe S has a much larger volume fraction of pearlite, which nucleates voids at higher temperatures more easily than the ferrite phase. Thus, because pipe S has more pearlite, voids can form more readily, and the upper-shelf energy is reduced. The earlier void initiation usually results in a larger dimple size for pipe S (Fig. 8). This behavior also matches that of the tensile ductility at higher temperatures where ductile fracture occurs.

It is apparent from the transition temperatures that neither of these steels will significantly resist fracture at very low temperatures. In fact, at liquid-nitrogen temperatures, these steels absorbed less than 3.5 J in the Charpy test, and the pipe L material fractured in a completely brittle manner during tensile testing. Whereas pipe S material had some impact resistance at room temperature, pipe L material had already reached the lower shelf. Thus, it seems clear that these materials will offer very little resistance to fracture during exposure to very low temperatures.

The possibility of fracture of the pipes during this method of repair will depend on the presence of flaws in the piping material, the stresses encountered during the operation, and the temperature. It is reasonable to believe that some flaws exist in the piping system, because it was installed many years ago and because some damage is to be expected during installation and operation of such a system. The temperatures during the repair operation will be sufficiently low to reach the lower-shelf regime, particularly in the area of the jacket, which will reach  $-196^{\circ}\text{C}$ . At these low temperatures, the materials are very brittle. The probable magnitude of the stresses during the operation is the critical issue of concern. These stresses are difficult to estimate. There may be residual stresses in the piping resulting from installation. Thermal stress may arise because of extreme variations in temperature, particularly if there are large thermal gradients. The cutting operation may cause vibrations and

impulse loads from tool chatter or alignment problems. The combination of these factors makes it appear quite possible that an existing flaw in material at very low temperature may be subjected to stresses and possibly impact loading. It is likely that fracture could occur; therefore, this technique is not recommended for these pipe materials. Caution is advised in applying this technique to any pipe with unknown toughness properties.

#### CONCLUSIONS

The mechanical properties and microstructures of two steel pipes removed from the ORNL chill-water system have been studied. The pipes were made of low-carbon steels, with microstructures of ferrite and pearlite. Pipe S had a small grain size and a banded microstructure, and pipe L had a larger grain size with less pearlite, randomly spaced. Pipe S had a DBTT of 0°C, compared to 84°C for pipe L. Because of the high transition temperatures and the very low energy levels of the lower shelves, the method of using liquid-nitrogen cooling would result in a small margin of safety for these pipes. Therefore, without more extensive stress analyses and nondestructive examination of the candidate pipes, this technique cannot be recommended for the type of pipe materials examined in this report.

#### ACKNOWLEDGMENTS

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