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CONSIDERATIONS IN THE EVALUATION OF GRAPHITIZATION IN PIPING SYSTEMS

Laboratory investigations of graphitized piping, sampling, bend and impact testing, and metallographic grading; interpretation of test results; number of specimens required; effects of heat treatments

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Introduction

Graphitization is still a problem of major concern to almost all power companies operating steam stations with carbon or carbon-moly piping, valves, headers, saddles, etc. New cases of serious graphitization are constantly found as a result of sampling programs in joints which had not been sampled previously as well as in joints which in earlier examinations appeared to be free of graphitization or contained only a mild, insignificant degree of graphitization.

In a number of instances potential failures in main steam piping joints have been avoided by a timely graphitization study. In several cases cracks have been found in the graphitized plane penetrating the wall thickness to within $^1/_4$ in. Since such cracks more commonly originate at the outside diameter, they are occasionally discovered by magnetic particle inspection techniques.

Laboratory exposure tests of carbon and carbon-moly steels at elevated temperatures have not provided a satisfactory correlation to the types of graphitization observed in steam piping systems under actual operating conditions. Thus, proper evaluation of graphitization can be made only on the basis of extensive experience gained over many years in the examination of power piping materials.

Graphitization only embrittles a joint. Actual failure requires additional stresses of considerable magnitude. In almost all cases, failures in service of graphitized piping have been associated with mechanical or thermal* shock or fatigue. Such failures have been the result of either one severe shock causing sudden rupture across the whole thickness of the joint or fatigue resulting in a somewhat slower initiation and propagation of a crack through the graphitized zone.

This complicates interpretation since there is no quantitative measure which determines the point at which the degree of graphitization (i.e., the embrittlement in the graphitized zone) is sufficiently severe to require repair of the joint by grooving out the heat-affected zone and rewelding. Under one set of operating conditions, as at locations in the pipe line far removed from the boiler or from other connections where sudden water carry-over resulting in a severe thermal shock is most unlikely, a heavier degree of graphitization might be assumed to be acceptable than at locations where water carry-over is a possibility.

Another complication is a lack of standardization in the testing procedures. Investigators vary somewhat in their sampling and testing procedures and in the interpretation of their findings.

The evaluation and discussion of the various testing criteria and of the results of several hundred commercial investigations is the major purpose of this paper.

Sampling

The procedure most widely used in determining the presence of graphitization in steam piping systems consists of removing boat-shaped slices from the welded joints by means of the so-called "weld-prober" saw illustrated in Fig. 1. The slice is taken across the weld and includes at least $^1/_2$ to 1 in. of base metal at each side of the weld. When properly prepared, the weld-probe slice provides sufficient material for a metallographic examination and, at least, one bend test, Fig. 2.

Pipe, valve or header sections of such dimensions as to make attachment of the weld-prober saw difficult may require special sectioning procedures as drilling or hack-saw cutting a wedged-shaped segment out of the joint. The cavities from which the weld-probe samples have been removed are usually rewelded and stress relieved to conform to ASA and/or ASME Code requirements.

Testing

Most laboratory evaluations of graphitized piping consist of cutting suitable specimens for metallographic

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* Thermal fatigue and thermal shock are terms used to denote the effects of temperature changes or alternating exposures at higher and lower temperatures on the life of a material. The differences between thermal fatigue and thermal shock are primarily related to the rate change of temperature and the severity of the temperature gradient. Thus, when the service life is primarily determined by the number of thermal cycles, failure is generally ascribed to thermal fatigue. However, when the severity of the temperature gradient or the rate of change in temperature is the primary cause of failure, the failure should be ascribed to thermal shock.[1]

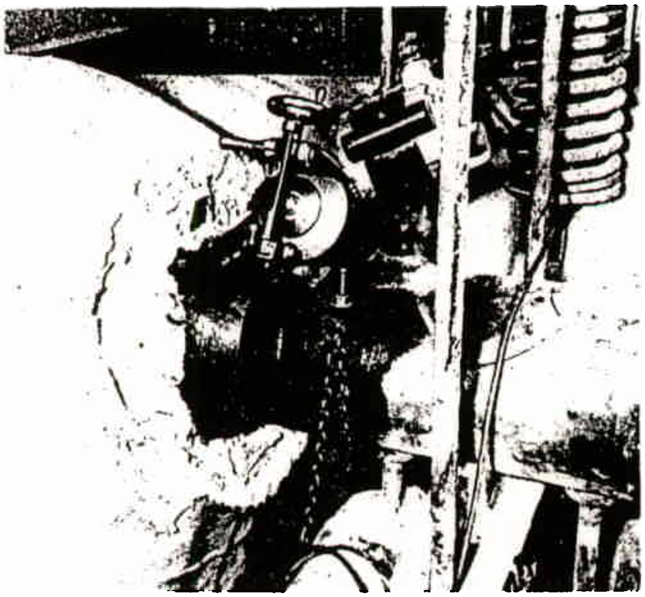


Fig. 1 "Weld-prober" saw in operation removing boat-shaped slice from main steam piping joint for laboratory evaluation

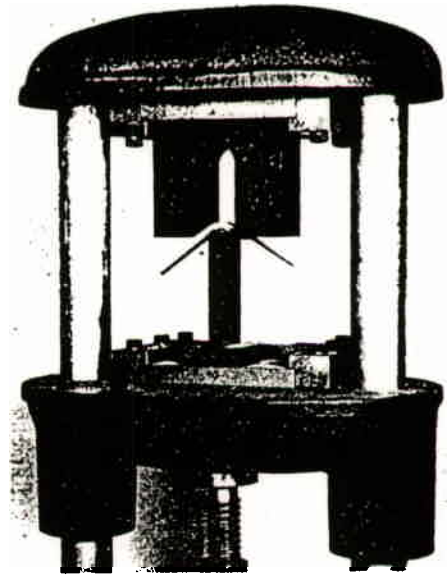


Fig. 3 Jig arrangement for guided bend test

examination and bend testing from the "weld-prober" segment.

No standard procedures have been established by any of the major technical societies concerned with power piping materials. Thus, there is some disagreement in the preparation of the test coupons, the testing procedures and the interpretation of the resulting test data as employed by different investigators. This increases the difficulty in comparing the results and interpretations obtained in different studies and/or by different investigators.

Bend Test

The slow-bend test is the mechanical test most commonly used to evaluate the level of graphitization in weld joints. Specimens generally are bent in a guided-bend test jig, as illustrated in Fig. 3.

There is considerable variation in the size of the specimens used. Specimen thicknesses may vary between $^1/_8$ and $^1/_4$ in. and widths between $^3/_8$ and $^1/_2$ in. Although the specimen length is not considered significant, a 3- to 4-in. length is most convenient.

Changes in the dimensions of the test jig introduce another variable. As a general rule it is recommended that the jig be designed to produce a bend radius of $r = 2t + {}^1/_{32}$ for specimens $^1/_3$ to $^1/_4$ in. thick[2] to $r = 2t + {}^1/_{16}$ for specimens $^1/_8$ to $^3/_{16}$ in. thick.

Bend-test results vary also with the location of the plunger in the jig in relation to the zone suspected of graphitization. Some investigators locate the pin perpendicular to the short axis of the specimen, i.e., the center of the plunger above the center of the zone suspected of graphitization as shown in Fig. 4*A*. Other investigators locate the plunger perpendicular to the plane suspected of graphitization as shown in Fig. 4*B*. The latter procedure represents the better practice and is followed by a majority of investigators.

Bending is usually continued until cracking occurs. In determining a quantitative value of the "degree" of graphitization some investigators use as criterion the angle of bend whereas others measure the elongation (usually in $^1/_2$ in.).

The bending angle or elongation may be measured when the first evidence of cracking is apparent, or after the crack extends across the width of the whole specimen. Many investigators measure the angle at the point where the crack has the initial length of $^1/_{32}$ or $^1/_{16}$ or $^1/_8$ in. Although it may be considered as arbitrary, a $^1/_{16}$-in. crack length is used now as the most satisfactory standard.

Because of the narrowness of the graphitized zone and the fact that the degree of graphitization may vary across the thickness of a joint, the proper evaluation of

Fig. 2 Sections for metallographic examination and bend testing cut from boat-shaped slice

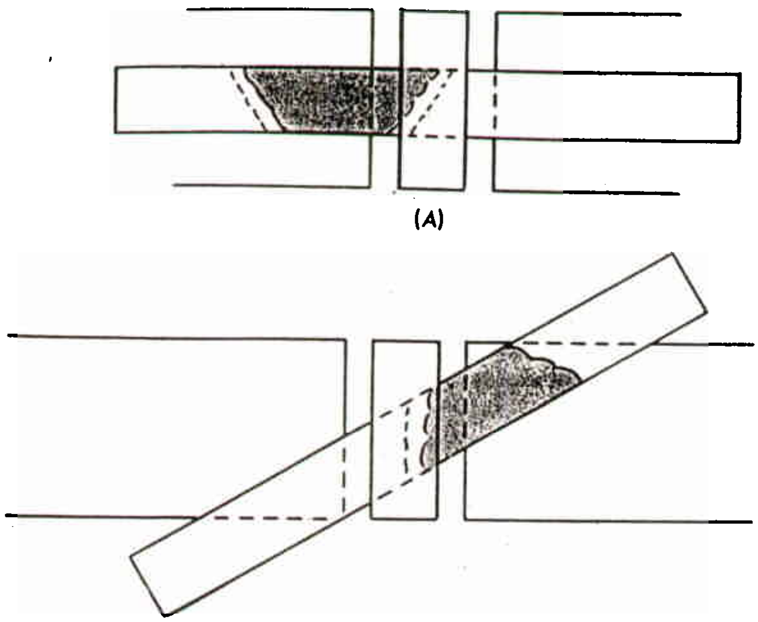


Fig. 4 Schematic sketch illustrating location of plunger in relation to graphitized plane. (A) Center of plunger perpendicular to short axis of bend specimen. (B) Center of plunger perpendicular to plane suspected of graphitization

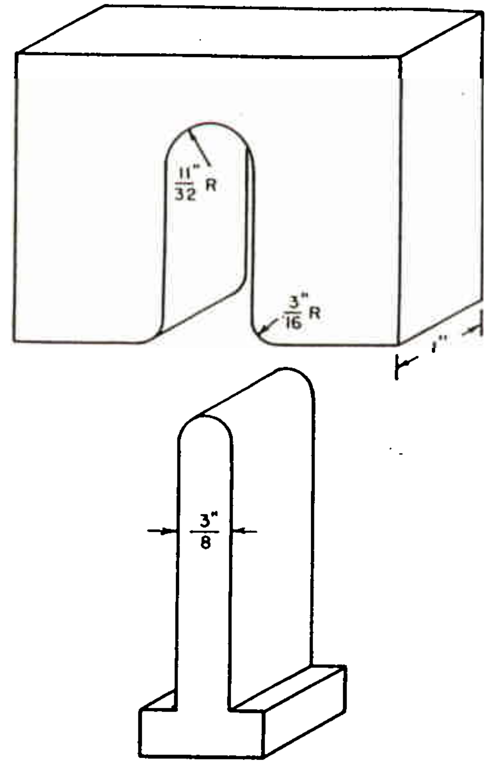


Fig. 5 Schematic sketch of standard test jig

these test variables is not readily made on graphitized specimens.

As an example, the scatter in test data of bend specimens across graphitized points is illustrated in Table 1 in which are evaluated the effects of variations in the dimensions of the test jig.

Table 1—Effects of Variations in Dimensions of Bend-Test Jigs Upon Bending Properties of Specimens* Removed from Graphitized Carbon-Moly Steel Piping

—Jig dimensions— Male (plunger) radius, in.	Female radius, in.	*Bend angle at point where initial crack extended* $^1/_{16}$ *in. deg.*	*Elongation at point where initial crack extended* $^1/_{16}$ *in., % in* $^1/_2$ *in.*
$^1/_4$	$^7/_{16}$	10	..
$^3/_{16}$	$^{11}/_{32}$	8	3
$^1/_8$	$^{11}/_{32}$	5	3
$^1/_{16}$	$^1/_4$	2	..
$^1/_4$	$^7/_{16}$	3	..
$^3/_{16}$	$^{11}/_{32}$	8	3
$^1/_8$	$^{11}/_{32}$	11	3
$^1/_{16}$	$^1/_4$	3	..
$^1/_4$	$^7/_{16}$	30	6
$^3/_{16}$	$^{11}/_{32}$	25	6
$^1/_8$	$^{11}/_{32}$	21	6
$^1/_{16}$	$^1/_4$	10	3

* Specimens $^1/_8$ x $^3/_8$ x $3^1/_2$ in.

The specimens in each of the three groups were from adjacent areas of three severely graphitized joints. The considerable scatter in the test results prevents any conclusions in regard to the size effect of the jig dimensions.

In order to evaluate these test variables properly and arrive at a suitable procedure, a more homogeneous material is necessary. Thus several types of steels were given various heat treatments to simulate the various degrees of brittleness found in graphitized joints. The specimens were heat treated uniformly and tested to isolate the various testing variables.

On the basis of these results, a jig was selected with a plunger (male), $^3/_{16}$ in. radius, and a female, $^{11}/_{32}$ in. radius, leaving $^1/_{32}$-in. clearance on each side of the specimen which is sufficient to prevent seizure of the bend specimen by the walls of the female member of the jig. A sketch of the jig is shown in Fig. 5.

A specimen thickness of $^1/_8$ in. was selected as most satisfactory, particularly since occasionally only relatively narrow weld-probe slices are removed from graphitized pipe joints. The specimen is illustrated in Fig. 6.

Since some investigators prefer to determine elongation whereas others believe bending angle is a better criterion of the degree of graphitization, measurements of both have been taken in our research and commercial investigations.

Relation between bend angle and elongation is illustrated in Fig. 7, determined at the point where the initial crack length measures approximately $^1/_{16}$ in.

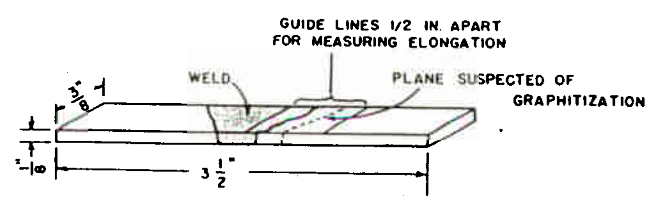


Fig. 6 Sketch of standard bend test specimen

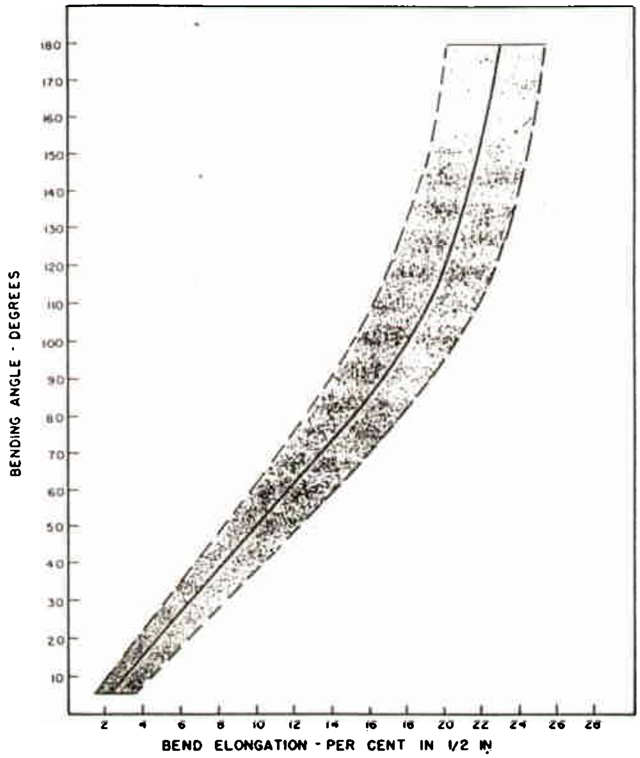


Fig. 7 Average relation of bending angle to elongation (determined at the angle where the initial crack length measures approximately $^1/_{16}$ in.)

The deviation from the mean curve may be as much as 20%. Since the possibility of error is somewhat greater in measuring the elongation than the bending angle, the latter seems the better criterion. It is preferred by most investigators.

Impact Toughness

Since embrittlement in steel is usually evaluated by means of impact tests, a series of tests was conducted to establish if any correlation could be obtained between impact and bend tests. Standard Charpy V-notch specimens were prepared with the root of the notch located in the graphitized plane as sketched in Fig. 8.

The results, plotted in Fig. 9, indicate that any correlation at best, is very poor. This may well be associated to the narrowness of the graphitized zone. As shown in Fig. 10, the fracture of several indentical specimens removed from the same joint and adjacent to each other did not follow the zone of graphitization. A severe embrittlement was indicated by the results of bend tests of specimens removed from the same joint.

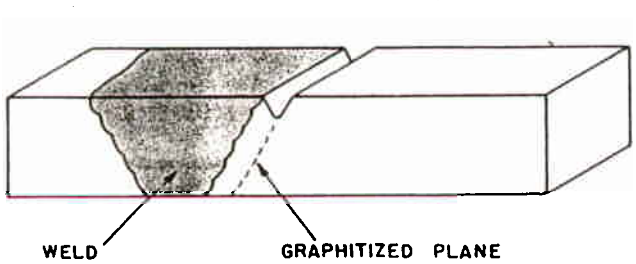


Fig. 8 Sketch of notch location in Charpy V-notch impact specimen

Metallographic Evaluation

Metallographic evaluation is most widely used to determine the degree of graphitization and to confirm the results of the guided-bend tests. The usual practice is to examine either the whole sectioned weld-probe specimen under the metallographic microscope or to cut smaller segments from this specimen representing heat-affected zone, base metal and weld metal. An initial examination is made at 100 diameters to determine the presence and amount of graphitization. The specimen is subsequently examined at 500 diameters to establish more clearly the degree and type of graphitization. Both magnifications are necessary for the proper interpretation of graphitization.

On the basis of 10 years of research in the laboratories of the Grinnell Co., a metallographic grading system of graphitization has been established which is illustrated in Fig. 11.[3]

The severity of graphitization depends upon the distribution, size and shape of the graphite particles. Whereas chain-type graphite, shown in Fig. 11*E*. usually indicates a "severe" or "extremely severe" condition, concentration of small nodular graphite particles (Fig. 11*F*) or a smaller number of very large nodular particles (Fig. 11 G) may be just as severe as the chain-type formation.

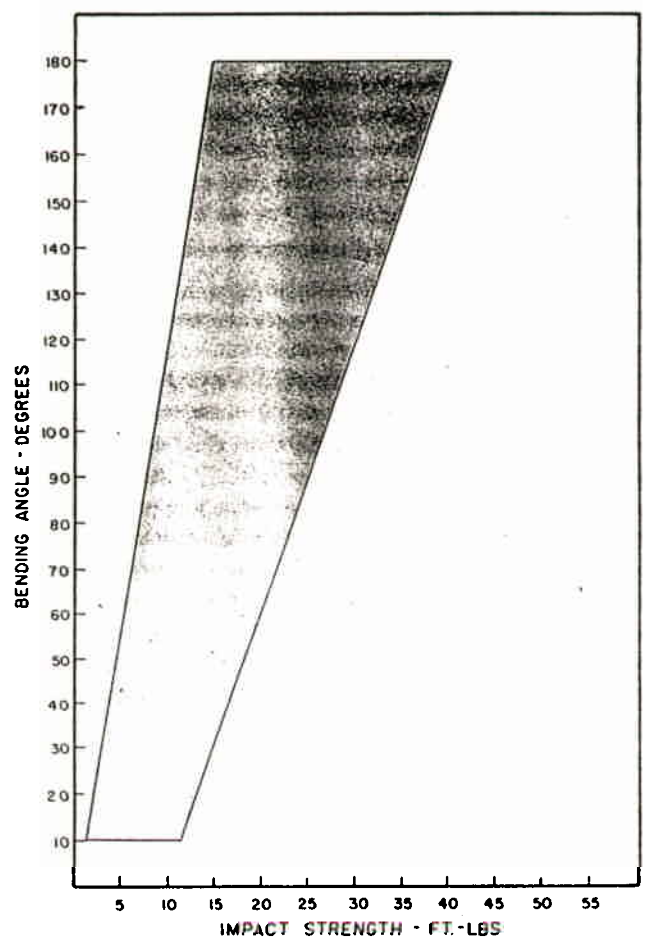


Fig. 9 Average relation between results of bend tests and impact tests. Specimens removed from adjacent areas in carbon-moly steel joints exhibiting different degrees of graphitization

The agreement between a careful metallographic evaluation and guided-bend tests is usually quite good. This is illustrated in Fig. 12 in which are summarized statistically the results of the various commercial investigations conducted during the past three years. By far the largest number of specimens exhibit either no graphitization or only very mild, mild or moderate degrees of graphitization. The microstructures shown in Fig. 11 normally correspond to the following bend angles:

Metallographic evaluation	*Bend angle, deg*
Very mild	Over 90
Mild	Over 90
Moderate	50–90
Heavy	30–50
Severe	15–30
Extremely severe	Below 15

Uniformity of Graphitization

One of the problems in setting up a sampling program is to decide on the number of samples which should be removed to provide a representative evaluation of a piping system.

A "safe" evaluation is considered by the Subgroup of the Metallurgy and Piping Subcommittee of the Edison Electric Institute[2] to be one in which three weld-probe samples are removed at 120-deg intervals around the circumference of each pipe or valve joint. Fewer weld-probe specimens are considered to be adequate only if the respective joint has been sampled previously and the variation in the degree of graphitization around the joint has been previously determined.[2]

Such extensive sampling programs are not always practical or economical. For example, where service conditions are such that graphitization is unlikely, a "spot" sampling program may be sufficient and serve as a preliminary basis for a more extensive program contemplated at some future date. To determine the variation in the degree of graphitization, approximately 1000 pipe-to-pipe and pipe-to-valve joints, sampled at two or three locations over the past five years in 30 typical steam stations, were analyzed statistically.

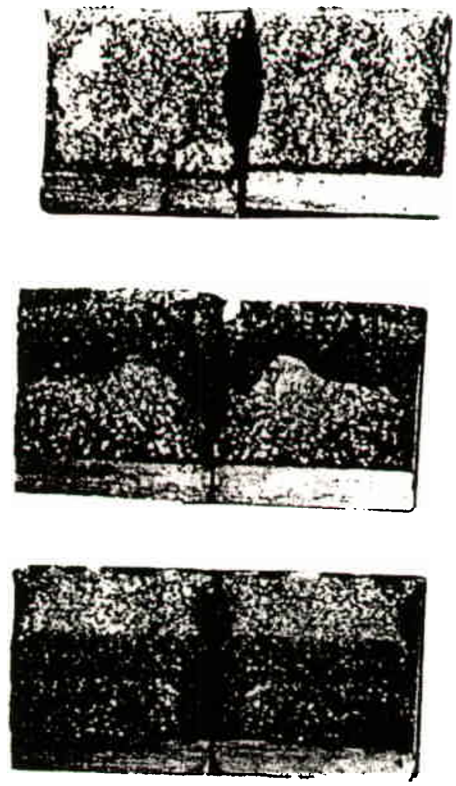


Fig. 10 Fracture surfaces of three Charpy V-notch impact specimens from adjacent areas of a severely graphitized carbon-moly valve steel

The results are summarized in Table 2. (Saddle, header, etc., are not included.)

Table 2—Variation in Degree of Graphitization in Carbon-Moly Steel Joints Sampled at Two or Three Locations as Determined by Metallographic Evaluation (None, Very Mild, Moderate, Heavy, Severe, Extremely Severe)

Structural component	*Separation in grading*					
	Same	*1 deg*	*2 deg*	*3 deg*	*4 deg*	*5 deg*
Pipe, %	85	10	4	1	0	0
Valve, %	75	12	8	4	1	0

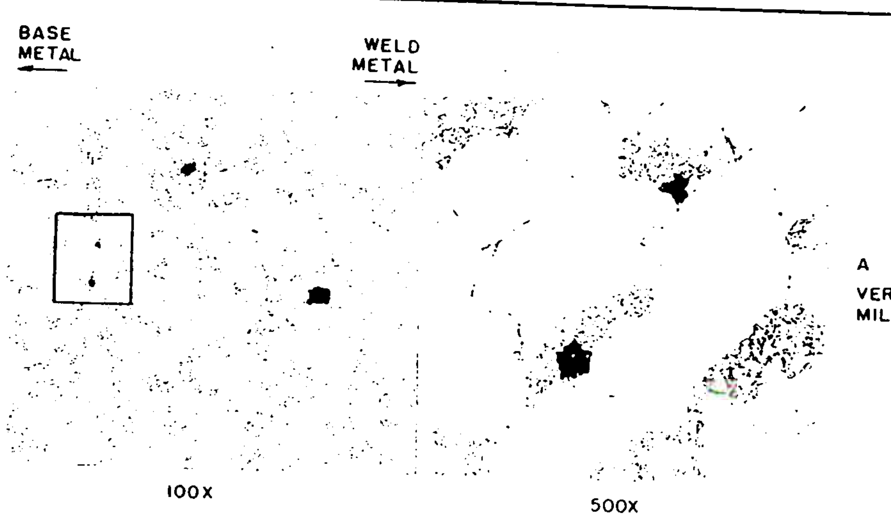
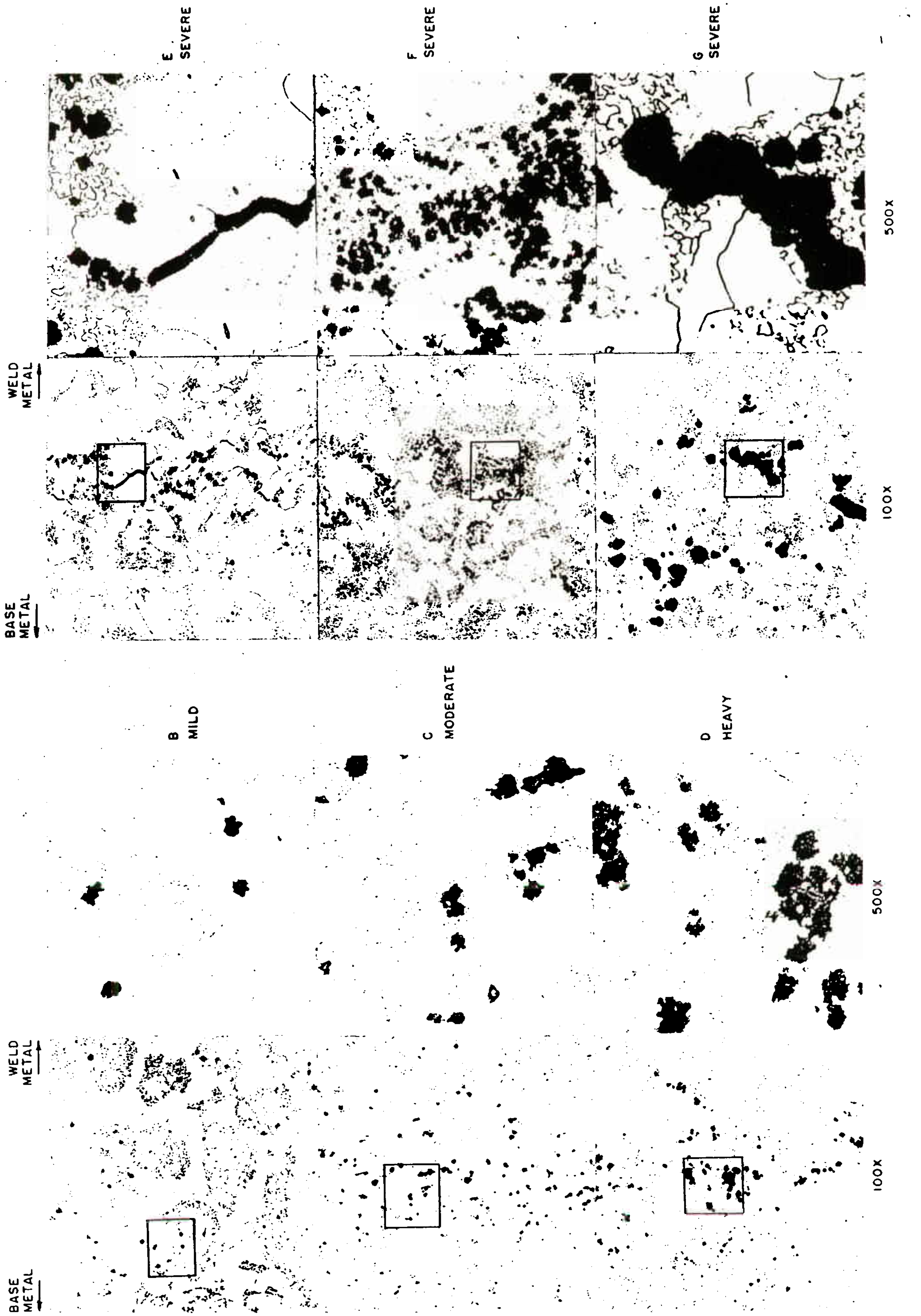


Fig. 11 Microstructures at 100 and 500 diameters illustrating various degrees of graphitization: (A) "very mild" graphitization; (B) "mild" graphitization; (C) "moderate" graphitization; (D) "heavy" graphitization; (E) "severe" graphitization; (F) "severe" graphitization; (G) "severe" graphitization. (Reduced by 1/3 upon reproduction)


BASE METAL
WELD METAL
B MILD
C MODERATE
D HEAVY
100X
500X
BASE METAL
WELD METAL
E SEVERE
F SEVERE
G SEVERE
100X
500X

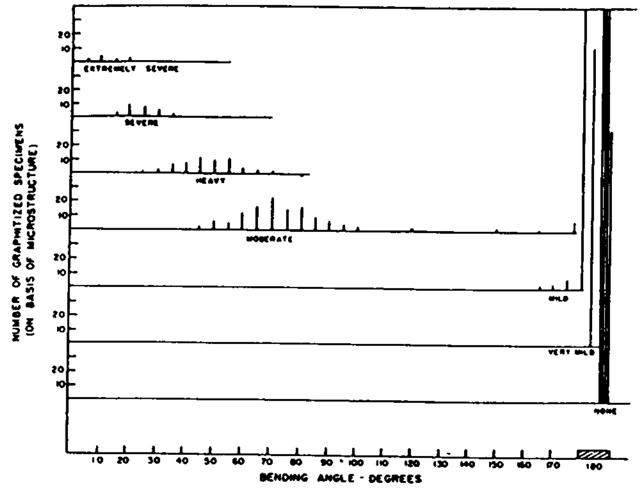


Fig. 12 Comparison of correlation between metallographic interpretation and bend test results of some 1700 weld-probe specimens

This shows that, in piping materials, 85% of the joints sampled by specimens at two or three different locations exhibited essentially the same degree (or absence) of graphitization around the periphery of the joint. In 10% of the joints the difference amounted to only about 1 deg; for example, the graphitization might have varied from none to very mild or from heavy to severe. In valve materials, the differences were somewhat greater.

If in this analysis all joints containing no graphitization are eliminated, the percentage variation increases, as summarized in Table 3.

Since the interpretation of the degree of graphitization often may be a borderline case, a 1-deg variation is not significant. Thus, these results show that in piping 15%, and in valves 30%, of the joints have a variation of two or more degrees which is significant; i.e., at one location the degree of graphitization may be "mild" whereas at another location the degree may be "heavy". An example of an extreme local variation is given in Fig. 13 illustrating "very mild" and "severe" degrees of graphitization in weld-probe samples removed 180 deg apart in a valve joint.

In some joints, the degree of graphitization varies also from the outside to the inside of the pipe or valve material. Where this variation occurs, the degree of graphitization tends to be more severe toward the outside diameter.

In most cases, the differences in the degree of graphitization probably can be associated with slight variations in the composition. This is particularly true in valves made of

Table 3—Variation in Degree of Graphitization in Carbon-Moly Joints Sampled at Two or Three Locations as Determined by Metallographic Tests. All Joints Containing at Least Some Graphitization (Very Mild, Mild, Moderate, Heavy, Severe, Extremely Severe)

Structural component	*Separation in grading*					
	Same	*1 deg*	*2 deg*	*3 deg*	*4 deg*	*5 deg*
Pipe, %	65	20	10	5	0	0
Valve, %	45	25	15	10	5	0

steel castings, which are more likely to exhibit greater variations in the composition than the hot-worked pipe steels. Moreover, castings, as a general rule, are likely to contain more carbon and aluminum than the corresponding grades of wrought and forged piping materials. In many foundries the 2-lb-aluminum-per-ton-of-steel addition needed for proper deoxidation is made in the runner box which, in rapidly solidified castings, may result in considerable variation in the deoxidation within the steel and a corresponding variation in the susceptibility to graphitization.

Differences in the stress, welding and postheating conditions may also have an effect upon the rate and resulting degree of graphitization. However, so far no satisfactory conclusion could be drawn as to the relative effect of each of these factors. The rate and type of graphitization also cannot be satisfactorily predicted

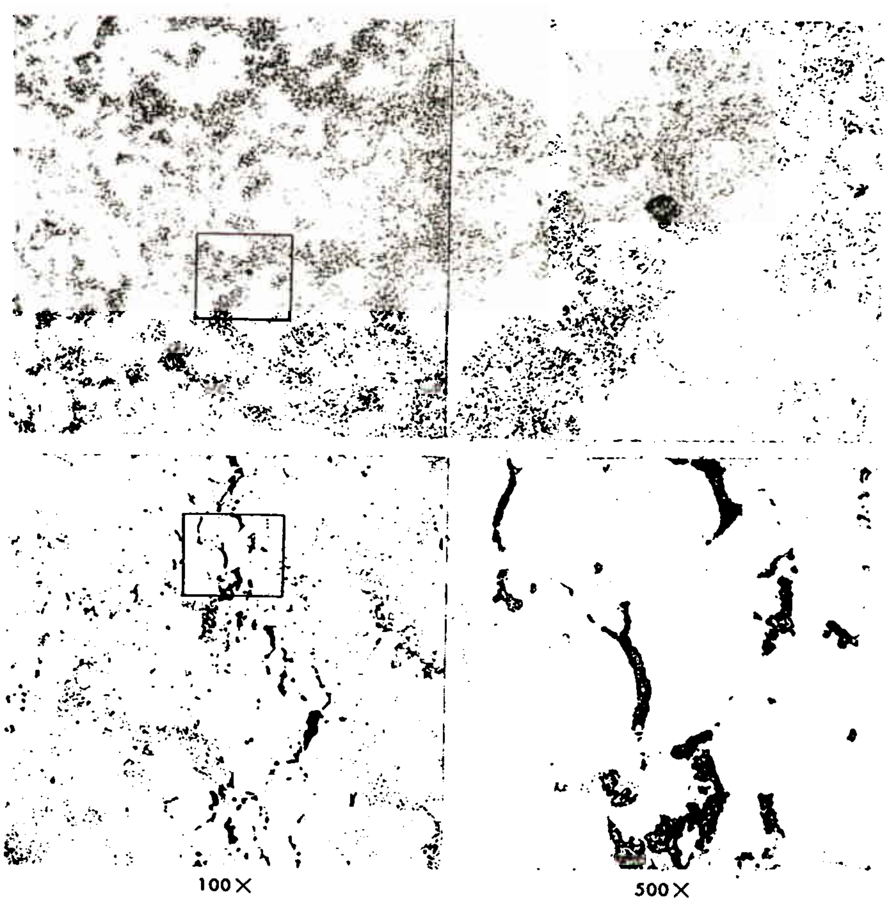


Fig. 13 Example of extreme variation in the degree of graphitization in two specimens removed 180 degrees apart from the same valve joint. (Reduced by $^1/_3$ upon reproduction)

on the basis of carbon content, deoxidation practice, grain size, heat treatment, welding procedure, stress, etc.

As a result, the removal of two "weld-probe" specimens from each pipe and valve joint seems highly advisable. However, in piping from the same heat or from similar heats where on the basis of a few previous samples it has been indicated that graphitization might be absent, one subsequent weld-probe specimen per pipe joint seems more than adequate. There are occasions when it is sufficient to examine only one end of a pipe. Moreover, as mentioned earlier, where service conditions are such that graphitization is unlikely, a "spot" sampling program may be sufficient.

A preliminary investigation may serve also as basis for a periodic sampling program in which a limited number of samples are removed and examined every few years to ascertain continued absence of graphitization. In such a program samples should be removed from a few previously sampled and unsampled joints. Limited sampling is also sufficient on piping produced by certain steel mills from particular heats that have a low-aluminum deoxidation practice which as the basis of previous experience has been found not to be susceptible to significant graphitization. Similar comments are applicable to the valve castings produced by certain valve manufacturers.

Heat Treatment

Various heat treatments are involved in graphitization studies. Some of these are postweld heat treatments whereas others are special heat treatments.

Postweld Heat Treatments

Where new welds are made, two types of postweld heat treatments are now widely used to inhibit subsequent graphitization in the heat-affected zone.

Most commonly used is a metallurgical "subcritical stabilization" heat treatment consisting of heating the joint between 1300 and 1350°F for a minimum of 4 hr. This heat treatment inhibits or, at least, retards further graphitization. Reoccurrence of graphite in carbon-moly steel joints, when heat treated in this manner, if at all, usually is in the widely scattered small nodular form which does not cause significant embrittlement.

Another post weld heat treatment consists of heating the joint at 1650 to 1700° F for 2 hr. This heat treatment is also believed to suppress graphitization. It seems possible, however, that where local induction heating is used, a plane susceptible to graphitization may form in the pipe outside the area covered by the induction coils where the temperatures for the induction heating operation reach 1350 to 1425° F. To date no information is available on the effects of this postheat treatment upon subsequent graphitization.

Special Heat Treatments

Another heat treatment occasionally used on graphitized piping is the solution heat treatment which consists of heating the graphitized area to temperatures above the upper critical (transformation) temperature of the steel. At these temperatures the "ferritic" steel has transformed into an "austenitic" steel in which the graphite will go into solution. Upon subsequent cooling, some carbon will remain in "solution" while the balance forms cementite particles. A commercial solution heat treatment consists of heating for 2 to 4 hr at temperatures between 1700 and 1750° F, depending upon the type and degree of graphitization. This is followed by controlled cooling to 1000° F at a rate of 300 to 400° F per hour.

Although several years ago this heat treatment was believed to have considerable merit for "rehabilitating" graphitized joints, the usefulness of this method is now very much doubted. Particularly where operating periods of five or more years are involved, the solution heat treatment provides, at best, a temporary cure. It is believed that graphite will reform more rapidly in the solution heat-treated, heat-affected zone than it did in the originally stress-relieved weld. It is also believed that the solution of graphite during the solution heat treatment leaves very small submicroscopic voids in the steel matrix. These voids continue to act as local stress raisers so that the solution heat treatment improves only slightly the ductility and toughness over that of the previously graphitized area. This is illustrated in Table 4, in which are given the results of bend and impact tests.

Table 4—Bend Test and Impact (Charpy V-Notch) Test Data of Graphitized Carbon-Moly Steel Material (85,000 hr at 950° F). Prior and After Solution Heat Treatment (1 Hr at 1700° F—Cooled 300° F/Hr)

Bend angle, deg				
As-received	54	59	86	56
Solution heat treated	84	86	105	165
Impact strength, ft-lb				
As-received	8.5	9.0	9.2	11
Solution heat treated	12	13	14	15

Only where a mild degree of graphitization is apparent and the graphite particles are relatively small does the solution heat treatment seem to have merit and may suppress further graphitization. Data on this are now being collected.

Since the solution heat treatment is made by local induction heating, the possibility of setting up a plane susceptible to graphitization some distance away from the induction coils must also be considered. As mentioned earlier, no information has been collected to date on the effects of such heat treatments. It may be that by following the solution heat treatment with a "subcritical stabilization" heat treatment, subsequent serious graphitization is avoided.

Conclusions

Periodic weld-probe sampling of carbon and carbon-moly steel piping systems in service at temperatures exceeding 800° F should eliminate hazardous conditions due to graphitization.

The elimination of significant degrees of graphitization, or the absence of graphitization, does not insure that a high-temperature, high-pressure piping system is safe. Other types of defects, completely unrelated to graphitization are occasionally found. Some have resulted in serious cracking which, in time, would have resulted in complete failure of the affected joint.

Careful periodic inspection of all piping systems is therefore advisable in order to maintain a proper operational level of safety.

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