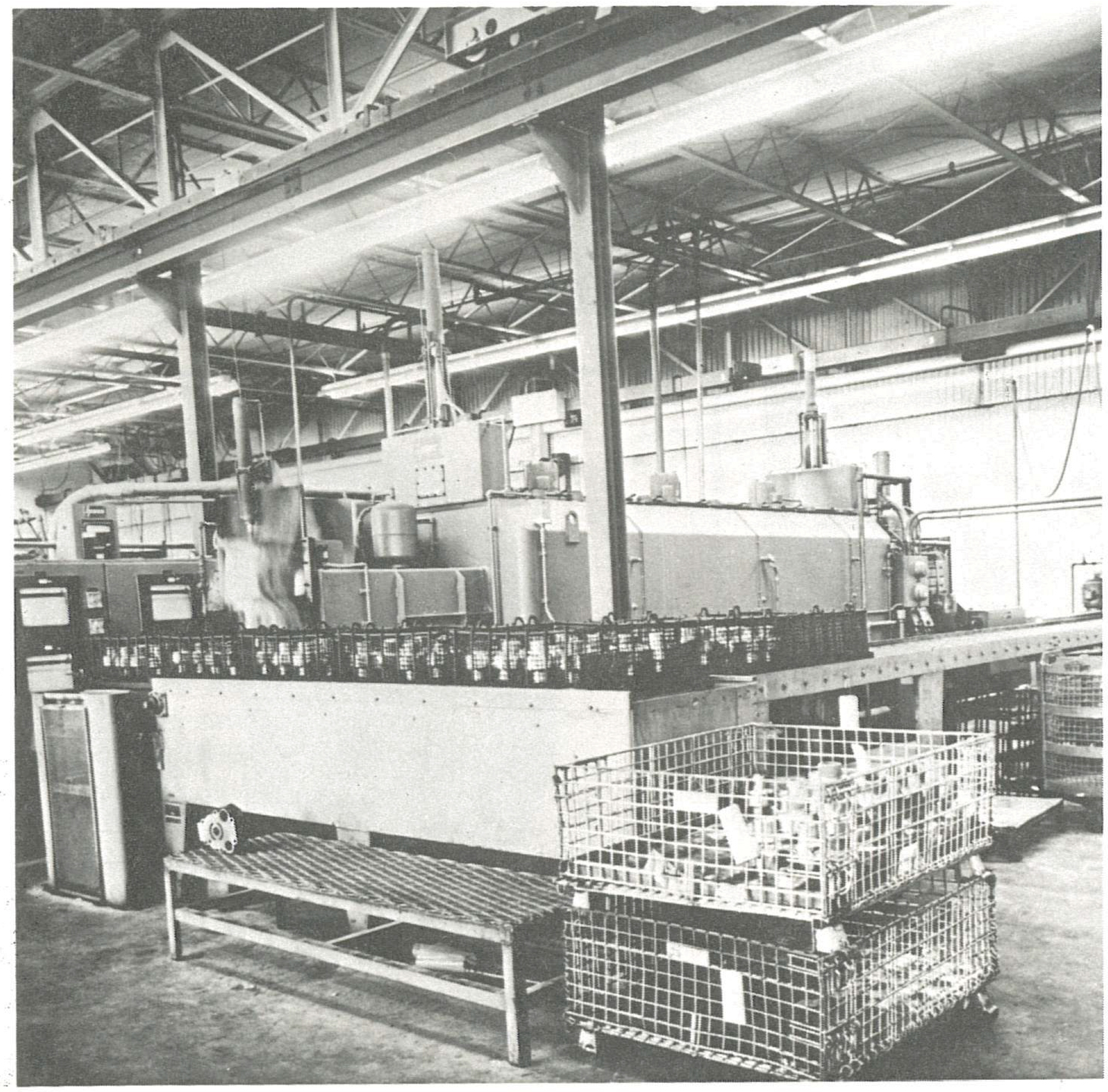
Nitemper Processing



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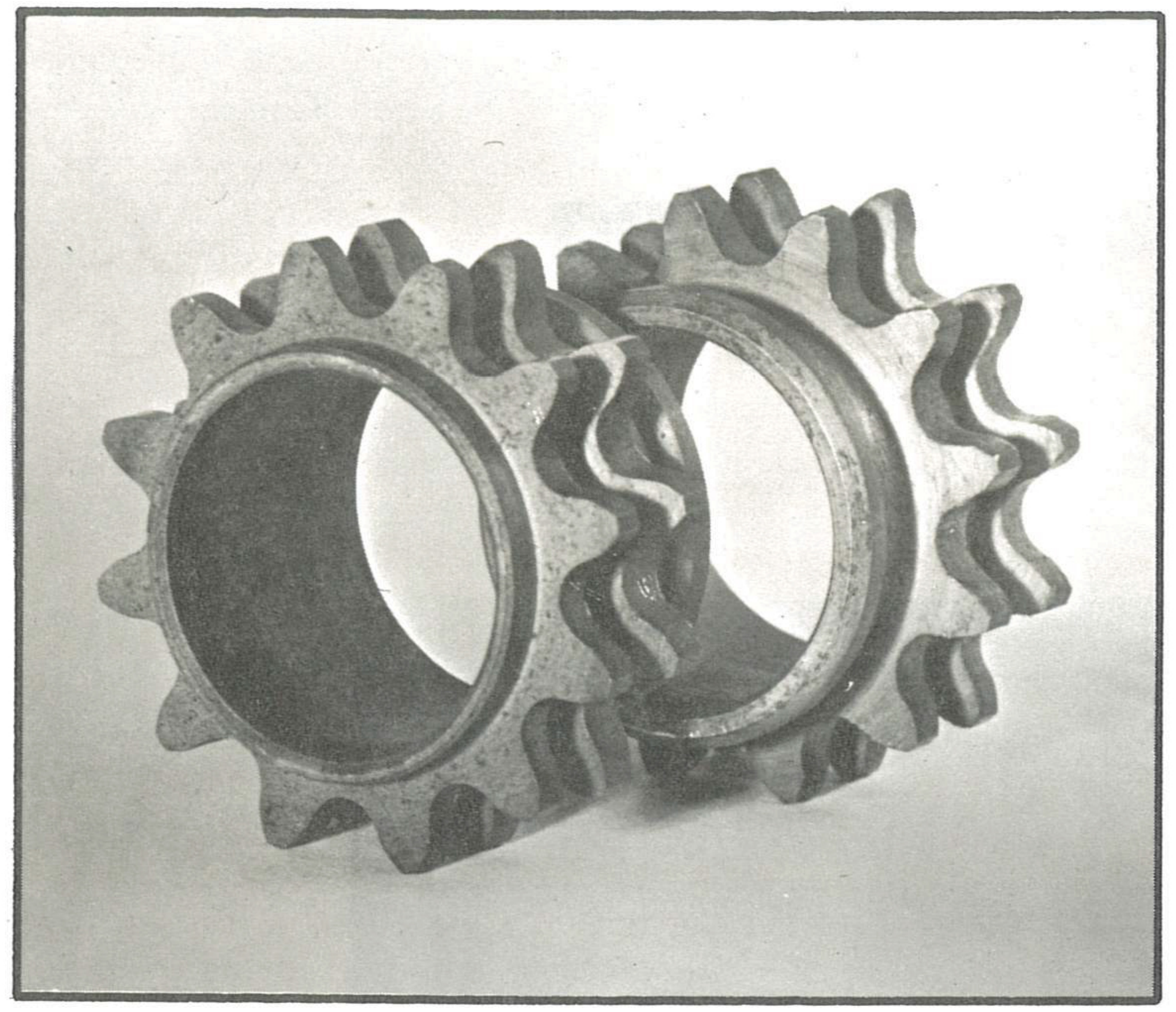
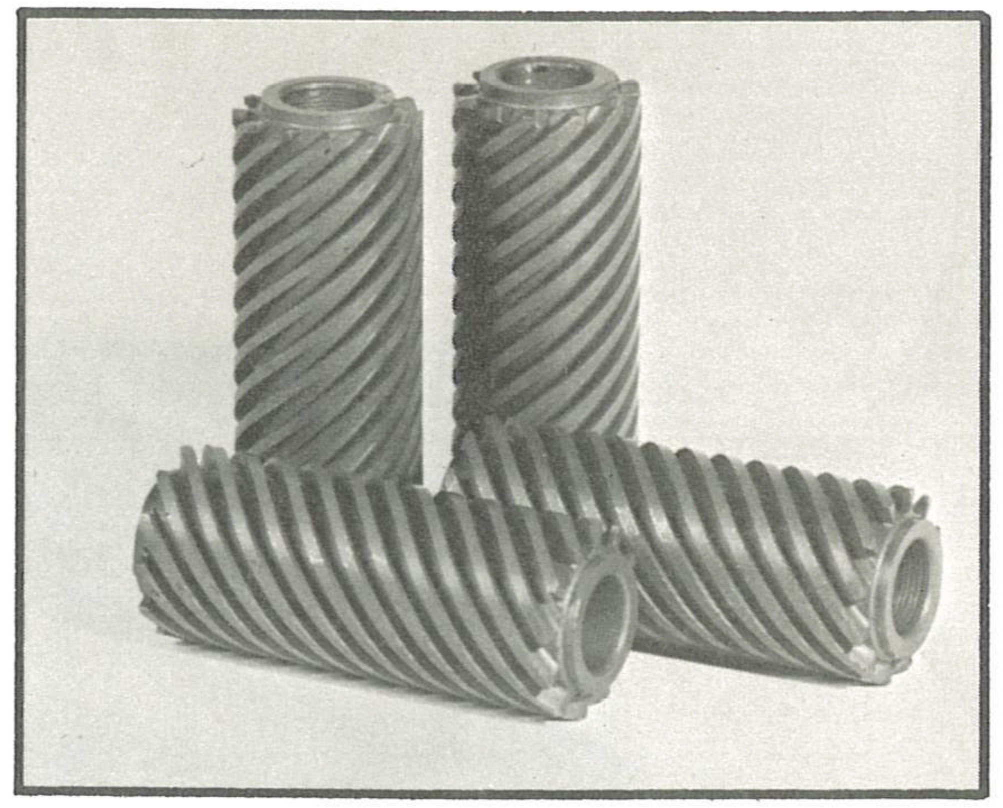
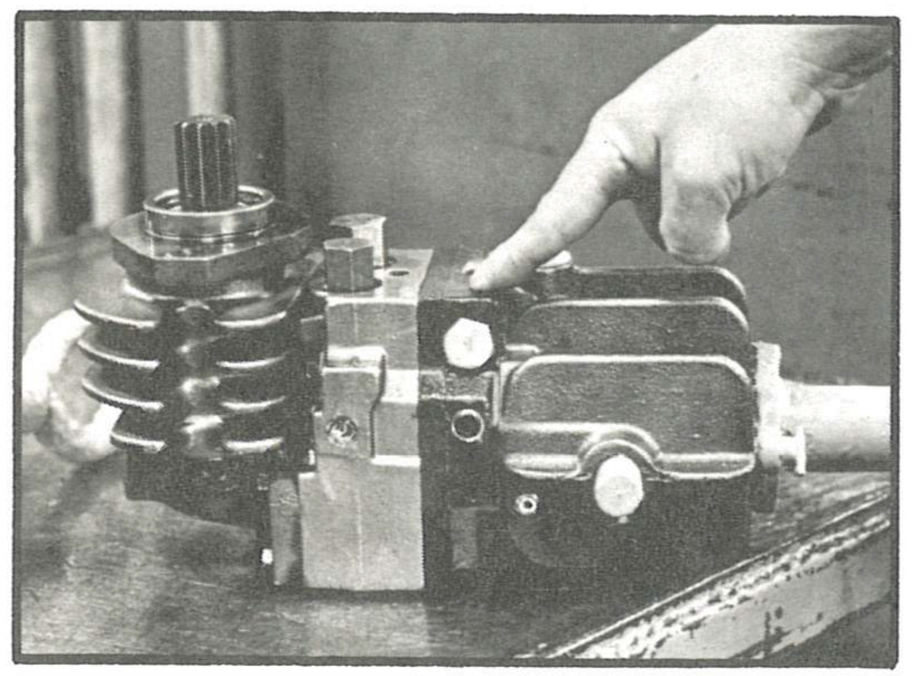
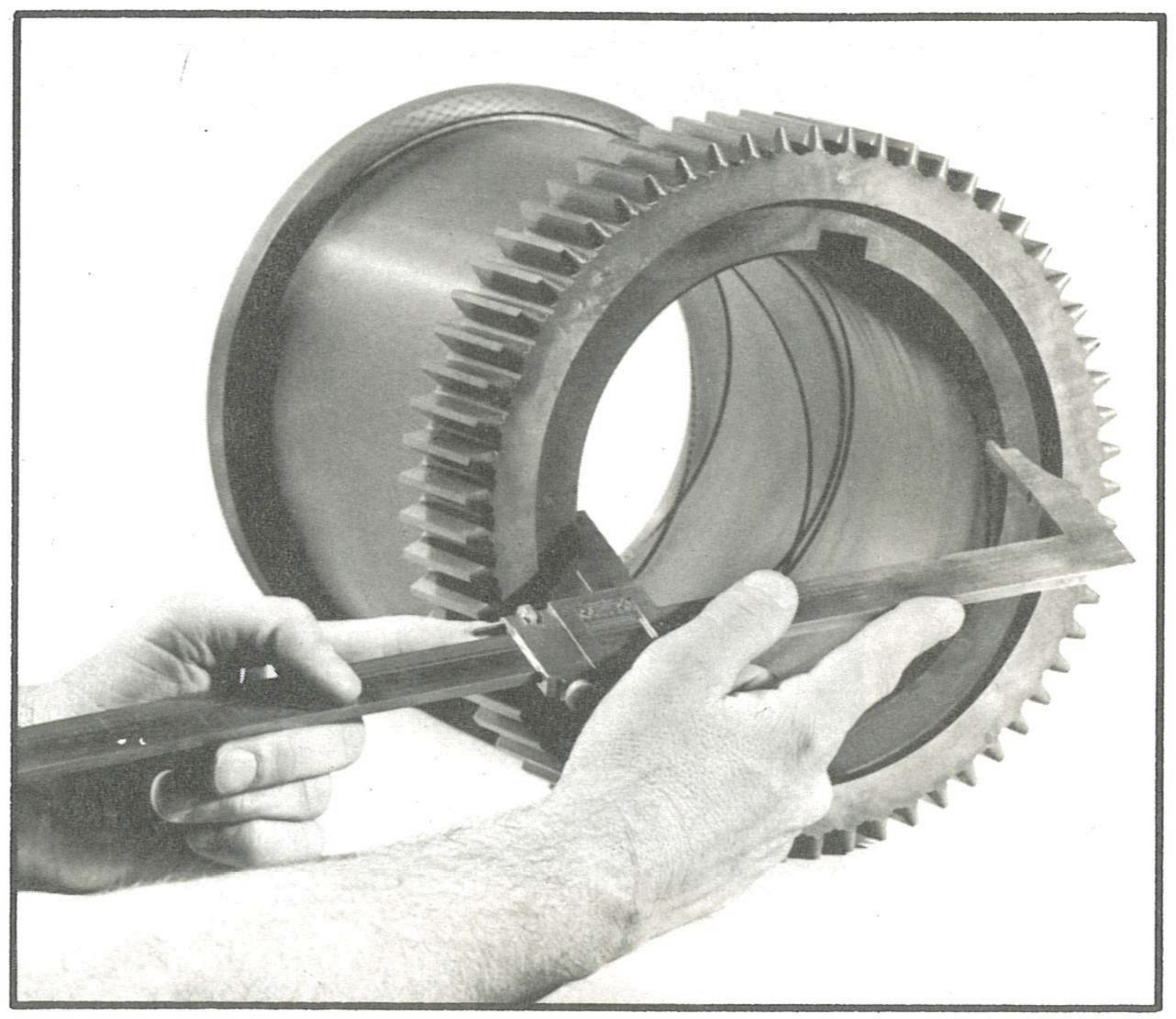
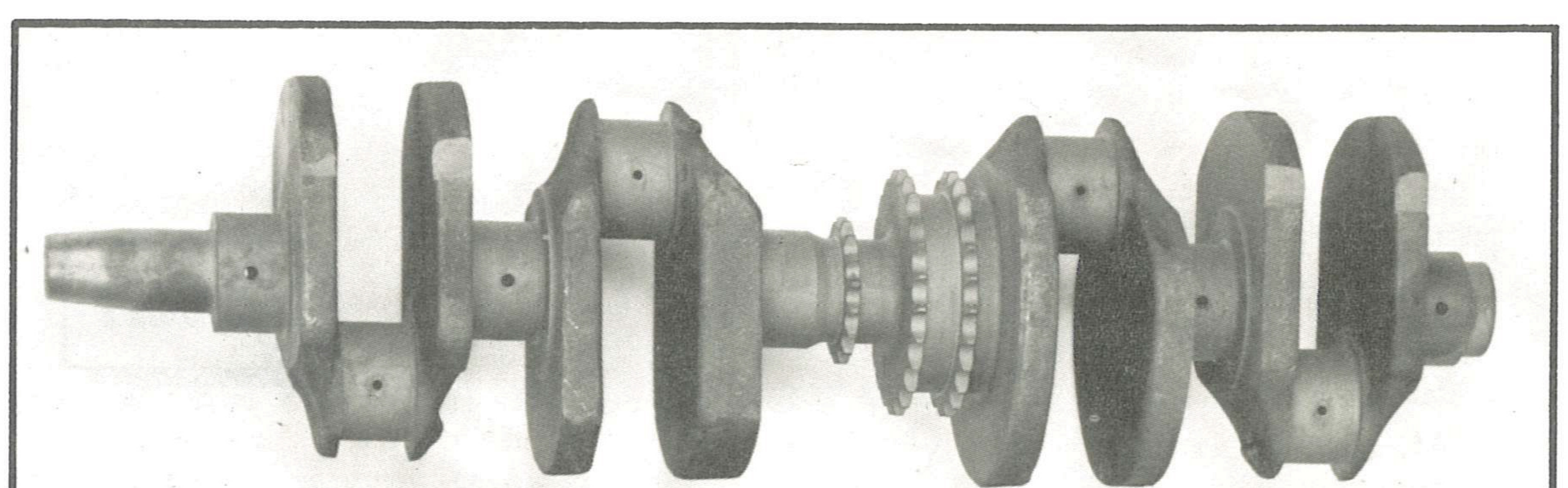










Nitemper PROCESSING

INTRODUCTION

In this era of rapidly-advancing metal technology, the demand for better performance characteristics has forced the metallurgical profession to develop improved metal processing techniques to keep pace with other segments of the metal-working industry. The needs of modern engineering for materials to facilitate reduced mass design and increased operating temperatures have frequently been answered by the development of new and improved case-hardening techniques for ferrous alloys. Ipsen Industries has recently perfected a modern new case-hardening method, the NITEMPER process. It offers many distinct advantages compared to similar case-hardening techniques already employed, particularly with regard to the quality of the resultant case and the simplicity of the processing procedure.

The development of the NITEMPER process is right in stride with some of the more urgent demands of our society, since it does not pose the pollution problems that are involved with its primary competitor, salt bath nitriding. This relatively simple atmosphere heat treating process does not require the exposure to or the elimination of poisonous and explosive chemicals and their toxic fumes.

NITEMPER processing is a low-temperature, controlled atmosphere heat treatment applied to steel and cast iron to obtain a tough case that greatly enhances wear and endurance properties. The process is carried out at a temperature below the transformation range for steel in an atmosphere consisting of partly dissociated ammonia and endothermic gas. A combination of properties, which are highly desirable for many engineering applications, are produced. These include:

1. A high surface hardness which is retained even after heating to as high as 1100°F.
2. Increased wear resistance — particularly for metal-to-metal wear.
3. Reduced tendency to seize and gall.
4. High resistance to fatigue.
5. A tough compact case with a reduced spalling tendency.
6. Minimum distortion resulting in low finishing costs.

The NITEMPER process should be considered for any applications involving wear or fatigue.

The NITEMPER process is simple, easy to carry out, and trouble-free if the precautions discussed later are observed. The process is performed by heating ferrous alloys of suitable composition in an atmosphere containing both a carbon and a nitrogen potential. The time required at the processing temperature of 1060°F (570°C) depends upon the steel being treated and the depth of case desired. The principal elements contained in solid solution in steel, which assist in the formation of useful nitrides which form a hard case, are aluminum, chromium, molybdenum, vanadium and tungsten. The hardening reaction occurs when atomic nitrogen from the dissociated ammonia diffuses into the steel and reacts with the alloying elements to form precipitates of hard alloy nitrides. The characteristic "white layer," developed on the surface of parts subjected to a nitriding operation, appears following formation of the nitrides. The nitrogen atoms remaining in solid solution serve to increase fatigue strength by blocking the development of potential fatigue cracks. The carbon derived from the carbonaceous atmosphere is relatively insoluble in steel at the processing temperature. However, the carbon potential of the processing atmosphere has a definite effect upon the nucleating characteristics at the surface of the ferrous alloy. It is considered to be quite tough and compact compared to the white layer developed by competitive processes.

A typical NITEMPER processing installation consists of an ammonia reservoir, an endothermic gas generator, proper equipment to monitor gas flow rates, and a well-designed furnace with close temperature control. The process is most readily performed with modified automatic, sealed quench furnaces. These units are suitable for other atmosphere heat treatments, such as carbonitriding, carburizing, hardening and tempering.

PROCESSING VARIABLES

The processing atmosphere, composed of 50% ammonia gas and 50% endothermic gas, is mixed and circulated within the furnace. The raw ammonia dissociates at the hot steel surfaces into atomic nitrogen and hydrogen according to the first stage of the following reaction:

$$2NH_{3(g)} \rightarrow 2N_{(g)} + 6H_{(g)} \rightarrow N_{2(g)} + 3H_{2(g)}$$

The atomic nitrogen, which is not readily dissolved by the ferrous alloy, and the atomic hydrogen pass rapidly through the second transformation of the above reaction. The resulting molecular forms are stable and inert, and must be exhausted from the furnace atmosphere. Dissociation rates of the raw ammonia, typically 50 to 60%, are influenced by the gas flow rates within the furnace. These dissociation rates should not be compared to those for other nitriding processes without considering that the processing atmosphere is diluted with an equal quantity of endothermic gas.

The carbon is provided according to the following carburizing reaction, as a direct result of the combustion of natural gas:

$$2CO_{(g)} \rightleftarrows C + CO_{2(g)}$$

Since endothermic atmospheres with a broad range of dewpoints are rich in carbon at temperatures in the range encountered in the NITEMPER process, close control of the dewpoint is not a requirement. Gas supplied by the endothermic generator for other atmosphere processes, normally in the +30°F dewpoint range, is suitable. Both the ammonia and the endothermic gas must be continuously replenished during the processing cycle to maintain the desired chemical reactions. The total volume of gas used during the NITEMPER processing cycle should approximate the total gas volume used during a corresponding carburizing cycle in a similar furnace.

The process temperature of 1060°F brings about the maximum diffusion rate of nitrogen while enhancing the carbide formation at the surface. A lower processing temperature may be used if the core hardness is a critical factor.

PRELIMINARY TREATMENT

NITEMPER processed parts do not warp nor distort to any appreciable extent, provided that the internal stresses resulting from machining and heat treating are removed before processing. Hardening, then reheating at 1100°F to 1300°F to obtain the desired core hardness promotes optimum response to processing and removes stresses produced by rough machining. Final machining or grinding is usually performed just prior to NITEMPER processing.

The surface to be processed should be a machined surface, clean and free from contamination. Any scale or decarburization due to earlier forming or heat treating operations should be removed; otherwise the NITEMPER case will be very brittle and will tend to spall. Stainless steels and other steels with high chromium contents rapidly form a chrome oxide compound layer on the surface, which prohibits the diffusion of nitrogen into the case. Vapor degreasing, abrasive cleaning, and electrolytic descaling are frequently used as surface conditioning treatments for these steels. A phosphate coating applied immediately after cleaning is commonly used to protect stainless steel surfaces from oxidation until NITEMPER processing can be accomplished.

CYCLE AND EQUIPMENT

Since the NITEMPER cycle durations are ordinarily only one-half to five hours, the modified automatic, sealed quench furnace lends itself most advantageously to the process. When a purge vestibule and quench chamber are incorporated in such a unit, the total furnace turn-around time can be significantly reduced. The purge vestibule is highly recommended since it greatly improves atmosphere control and utilization, while at the same time eliminating purging time in the processing zone.

The parts can be either oil-quenched or atmosphere-cooled in the quench chamber. Oil quenching promotes better endurance properties by (a) retaining larger percentages of the nitrogen of the diffused layer in solid solution and (b) retaining a higher compressive stress level in the case. It is commonly accepted theory that fatigue failures originate slightly below the surface in conventionally case-hardened parts. This would correspond to the diffusion zone in a NITEMPER processed case. In all probability, the nitrogen atoms suspended in solid solution in the ferrous matrix lock the slip planes blocking the development of potential fatigue failures. Quenching also prevents to a large degree the reduction of stresses inherent with slow cooling. This is important, since the comprehensive stresses developed in a hardened case proportionately increase the critical tensile stress required to cause fatigue failure of this material. Atmosphere cooling is recommended when distortion is extremely critical. The total absence of sooting results in clean quality work as evident in Figure 1 (see inside front cover).

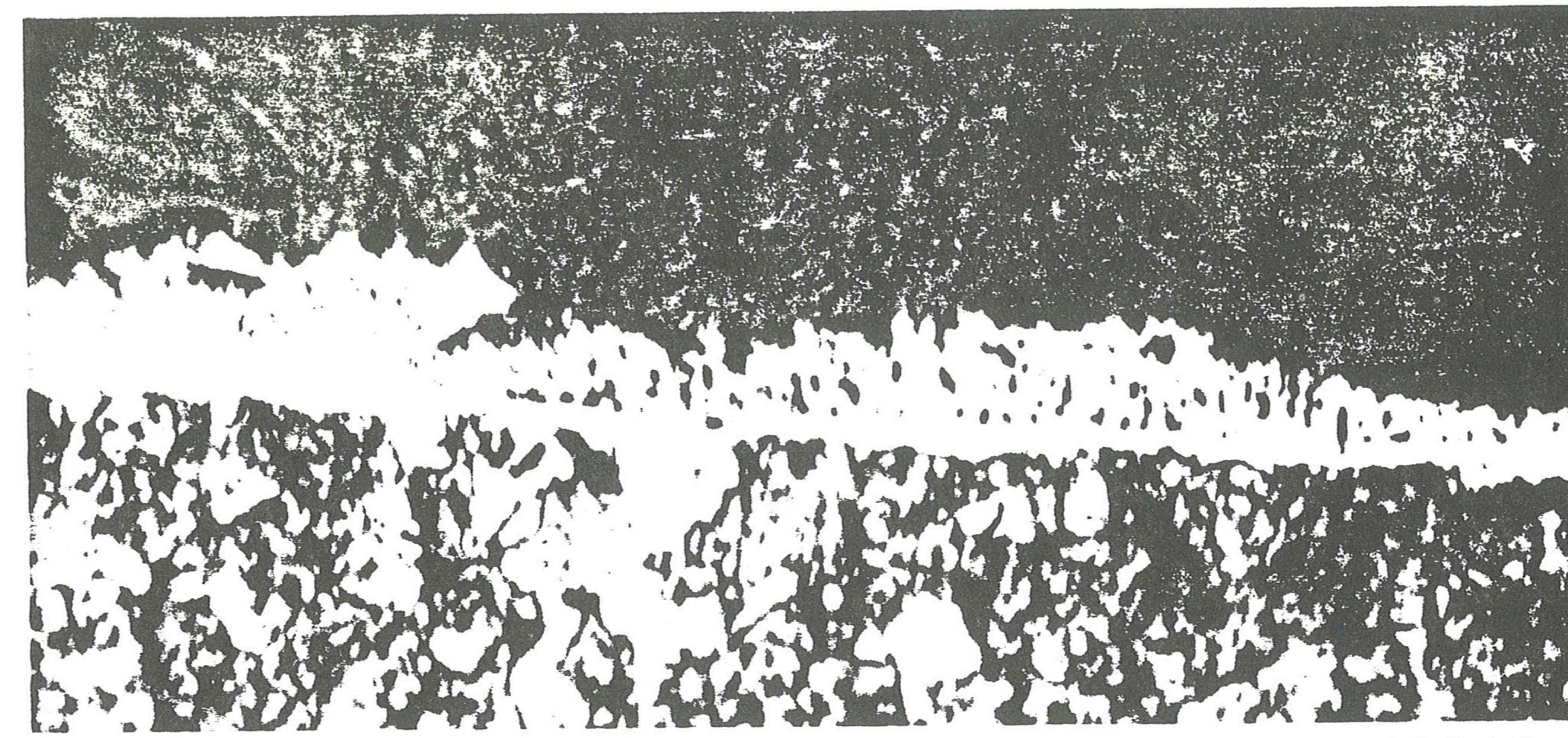
Forced convection heating is essential in furnaces used for the NITEMPER process.

This feature, along with adequate gas flow rates, assures the exposure of all work surfaces to the processing atmosphere. Dead spots and non-uniform temperatures result in uneven case depths. A gas-tight furnace enclosure is necessary to make sure that the ammonia and endothermic gases are exhausted through the effluent vent where they are burned.

The refractory in the atmosphere furnace should preferably be mullite rather than silicon carbide, since the latter material deteriorates after prolonged exposure to the ammonia atmosphere at the lower temperature. Either of these materials can be used for higher temperature atmosphere applications. The furnace being used for conventional carburizing can be conditioned for the NITEMPER process in less than one-hour's time. Utility and flexibilty of the processing equipment are an advantage offered by the NITEMPER process.

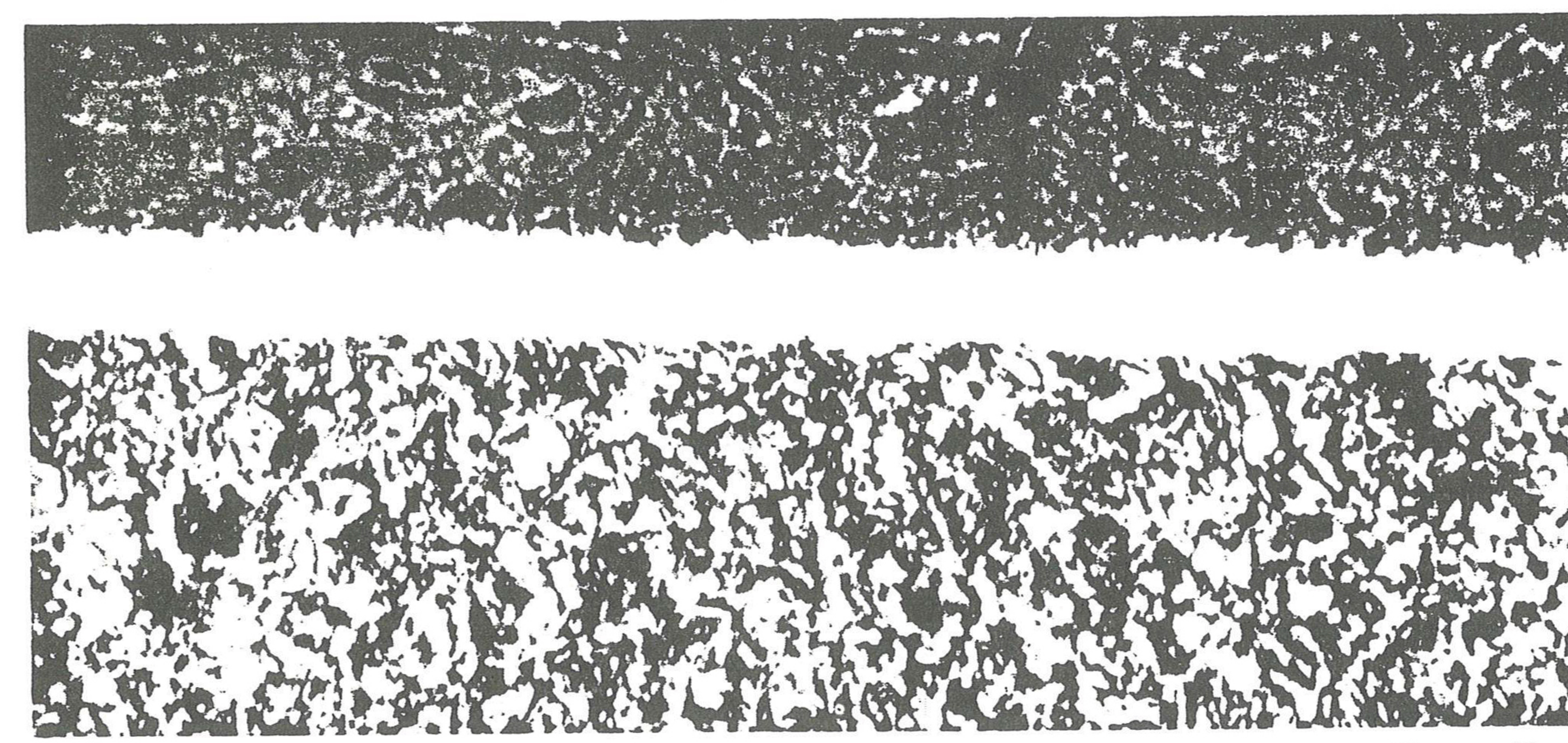
If the precautions ordinarily observed when dealing with any flammable furnace atmosphere are also observed when NITEMPER processing, operating hazards are negligible. The usual care should be taken to immediately ignite any gases valved into the furnace, maintain flame curtains at the external extremities of the furnace, and operate with a positive furnace pressure throughout the cycle. A furnace designed for performing the NITEMPER process is shown in Figure 2 (front cover).

FIGURE 3



Salt Bath Process

FIGURE 4



Nitemper Process

METALLURGICAL PROPERTIES

As previously pointed out, the NITEMPER processing atmosphere provides both carbon and nitrogen to the surface of the ferrous alloy. Nitrogen is more soluble than carbon at these temperatures and diffuses into the work while the carbon forms iron and alloy carbide particles at or near the surface.

The NITEMPER processed case consists of the characteristic "white layer" common to all nitrided parts and the underlying diffused layer. The properties of the white layer, typically .0001" to .0015" thick, are critical for most applications. It is considered to be quite tough and compact compared to the white layer developed by competitive processes. The reason for this is believed to be closely related to the rich carbon potential of the processing atmosphere and its effect upon the nucleating characteristics at the surface of the ferrous alloy. The nitrogen within the white layer precipitates out of solid solution as either alloy nitrides or carbon-bearing epsilon iron nitrides. The amount of the brittle Fe_2N phase formed is substantially reduced, compared to the results obtained in conventional gas nitriding. The reduced proportion of this brittle iron nitride phase in the white layer appears closely related to the effects of the carbon. This theory is substantiated by the fact that the white layer produced by salt bath nitriding has a notably reduced tendency to spall compared to the white layer produced by gas nitriding. The processing media for both salt bath nitriding and the NITEMPER process contain nitrogen as well as carbon, while the gas nitriding process utilizes only nitrogen. The NITEMPER process produces a white layer with comparatively the same degree of toughness and ductility as salt bath nitriding. However, one distinct difference is that the NITEMPER process produces a much more compact and more uniform white layer than salt bath nitriding. (Refer to Fig. 3 and Fig. 4.)

The nitrogen which diffuses through the white layer into the diffusion layer beneath is either precipitated as alloy nitrides or retained in solid solution. The formation of the precipitated nitrides is responsible for the hardening mechanism in the diffusion layer as it is in the white layer. However, the degree of hardening is reduced because of the diminishing concentration of diffused nitrogen. The effect of the nitrogen retained in solid solution in the diffusion layer upon the fatigue strength has already been discussed.

Relative values of the white layer depth with respect to diffusion layer depth, as well as the absolute values of each. depend upon (1) processing temperature, (2) processing time, (3) content of nitride forming elements and, (4) combined carbon content. All must be considered, but for a given set of parameters, the values are readily controlled and can be accurately predicted.

Like most diffusion-related processes, the diffusion depth increases proportionately with time. Temperature is also a factor; the maximum diffusion rate occurring at approximately 1060°F with a reduced rate resulting at temperatures up to the transformation temperature for the steel. Higher percentages of carbon, nickel, and silicon dissolved in the ferrous alloy also reduce the nitrogen diffusion rate. The influence of carbon is the most drastic.

Aluminum, chromium, molybdenum, vanadium and tungsten are the alloying elements commonly found in ferrous alloys which form stable nitrides. With increasing percentages of these elements in solid solution, the precipitation of a large proportion of the nitrogen as nitrides will reduce its diffusion rate considerably.

Aluminum forms the hardest nitride, and for this reason is commonly found in steels developed for nitriding. Chromium, molybdenum, vanadium and tungsten form nitrides which are also relatively hard. The total content of these elements in the alloy will determine the maximum attainable hardness as well as the degree of reduction of the nitrogen diffusion rate. The influence of several alloying elements and the carbon content are apparent in Table 1. The resultant hardness profile and depth of white layer are indicated for a variety of steels commonly being NITEMPER processed.

APPLICATIONS AND RELATED ADVANTAGES

The NITEMPER process can advantageously be substituted for such case hardening techniques as salt bath nitriding, gas nitriding, carbo-nitriding, carburizing, and induction hardening. Typical components which have been successfully NITEMPER processed include camshafts, connecting rods, corrugating rolls, crankshafts, die casting dies, gears, retaining rings, shafts, splines, and wear plates. Steels from almost every classification have been NITEMPER processed. Field performance tests, wear tests, and fatigue tests often involving direct comparisons between similar components processed by other case hardening techniques, have been reported. The results have been most favorable, particularly when the NITEMPER processed components have been compared to salt bath nitrided components.

The NITEMPER process, compared to salt bath nitriding, offers the following advantages:

1. **Economy** — There are no additional costs for chemicals, license fees and special equipment.
2. **Pollution Control** — Fumes and salt disposal problems are eliminated.
3. **Surface Integrity** — There is no pitting, contamination or porosity due to a lack of process control.
4. **Safety** — Explosive and poisonous chemicals are not used.
5. **White Layer** — The resultant white layer is more uniform and compact.
6. **Corrosive Residues** — Processed parts do not require further cleaning — particularly parts with blind holes and intricate design.
7. **Technical Supervision** — The need for trained people to rectify and control salt baths is eliminated.
8. **Higher Production Rates** — The process is more flexible and adaptable to varying production requirements.
9. **Stop-offs** — The gas processing method more readily facilitates selective hardening treatments.

4 Hr. NITEMPER at 1060°F (570°C)								
Steel Sample * Hardness converted in Rc								
Depth"	1	2	3	4	5	6	7	8
Surface	71	58	62	63	69	70	68	69
.001	70	44	53	55	67	68	61	64
.002	70	37	54	55	66	67	56	58
.003	69	30	51	50	62	66	55	50
.005	64	26	51	48	50	63	55	41
.007	45	—	49	45	—	63	—	—
.010	36	24	47	35	—	—	—	30
.015	—	23	40	30	—	—	—	—
.020	—	22	30	—	—	—	—	—
Core	30	20	24	25	45	60	55	27
White Layer"	.0001	.0009	.0007	.0006	.0004	.0002	.0006	

* 1	Nitralloy N	.25C 1.15CR 1.00A1 .55Mn 3.5Ni .25Mo
2	1015	.15C .50Mn
3	8620	.20C .50Cr .20Mo .80Mn .60Ni
4	4140	.40C 1.00Cr .20Mo .90Mn
5	H13	.35C 5.00Cr 1.00V 1.50Mo
6	M2	.80C 5.00Mo 6.00W 4.00Cr 2.00V
7	D2	1.55C .30Mn 12.00Cr .90V .80Mo
8	410 Stainless	.10C .30Mn 12.50Cr

- All samples were hardened, tempered and then NITEMPER processed for four hours.
- The surface hardness was measured by determining the Knoop hardness number on a buffed processed surface which was machined to a fine finish before processing.
- All hardnesses were converted to Rc after being measured with a Tukon Microhardness Tester, using 500 gram load.

Table 1

10. **Modern Method** — The process is more compatible with and realizes more of the advantages of the modern heat treating facility employing automated integral quench furnaces rather than salt pots.

Compared to gas nitriding the NITEMPER process offers the following advantages:

1. **Tough Ductile Case** — The carbide formation promotes a tough ductile white layer which has a reduced tendency to spall.
2. **Less Processing** — The ductile white layer does not ordinarily require grinding.
3. **Short Cycles** — A satisfactory case is produced with minimum furnace turn-around time.
4. **Applications** — Reduced spalling tendencies allow it to be used on a greater selection of ferrous alloys.

The advantage most commonly realized when the NITEMPER process has been substituted for carburizing, carbonitriding or induction hardening has been less distortion and growth. This results in minimal finishing costs. Often a material can be up-graded by the NITEMPER process or an improved product can be made at an equivalent or even a reduced cost.

The NITEMPER process is very rapidly being accepted to improve wear and fatigue properties of ferrous alloy components. It is a modern economical process compatible with and designed for the requirements of modern industry.

IPSEN INDUSTRIES—Rockford, Illinois

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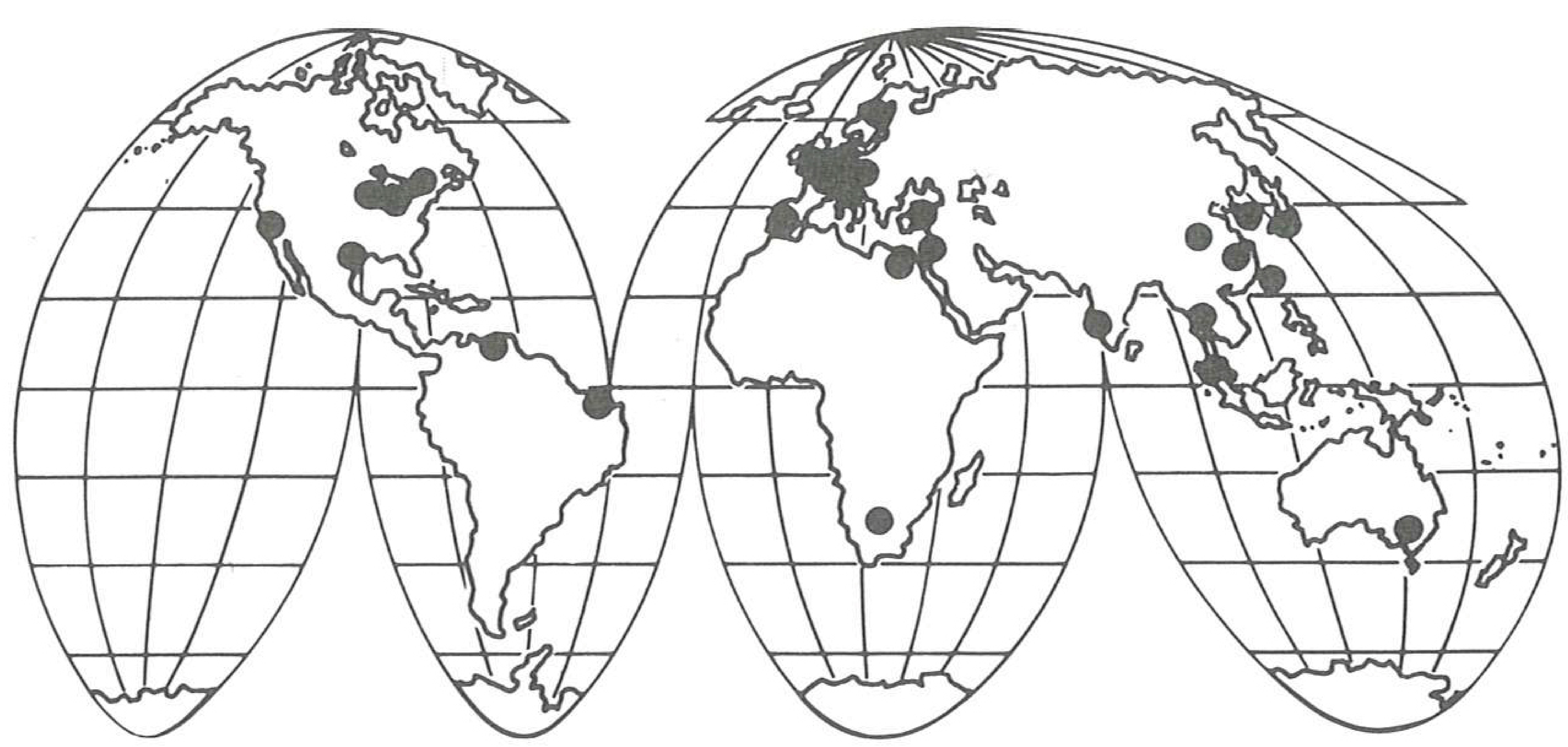

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